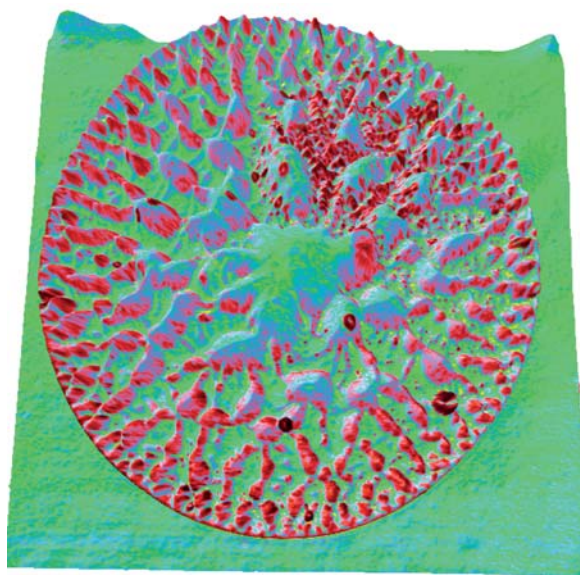




# PeakForce QNM

## User Guide



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### Document Revision History: PeakForce QNM User Guide

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BioScope™  
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**Software Modes:**

TappingMode™  
Tapping™  
TappingMode+™  
LiftMode™  
AutoTune™  
TurboScan™  
Fast HSG™  
PhaseImaging™  
DekMap 2™  
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**Miscellaneous:**

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**Cover Image:** Anti-bacterial film consisting of poly(methyl methacrylate) and silver nanoparticles. The sample was imaged on a Dimension Icon using PeakForce QNM at a scan size of 13.5 µm. The data shown is adhesion data overlaid on topography. The dark red spots correspond to the location of silver nanoparticles, which are difficult to identify using topography alone. (Sample courtesy of Mishae Khan and Daniel Bubb, Rutgers University.)

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# Chapter 1 Introduction to PeakForce QNM Microscopy

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## 1.1 Introduction

PeakForce QNM (Quantitative NanoMechanics), an extension of Peak Force Tapping<sup>TM</sup> mode, enables quantitative measurement of nano-scale material properties such as modulus, adhesion, deformation and dissipation. Because Peak Force Tapping Mode controls the force applied to the sample by the tip, sample deformation depths are small and the effect of the substrate on the measured modulus is decreased.

PeakForce QNM can provide compositional mapping of a complex composite material while providing equal or higher resolution than a TappingMode image (~5nm). Peak Force Tapping Mode has high spatial resolution, relatively high speed, and can detect a large range of elasticities. PeakForce Tapping mode produces similar results to HarmoniX (see the *HarmoniX User Guide*, Bruker p/n 004-1024-000 for details) but is much easier to use and covers a wider modulus and adhesion range.

With a calibrated cantilever, Peak Force QNM is quantitative and has high spatial resolution.

Peak Force Tapping Mode<sup>TM</sup> microscopy, the core technology behind PeakForce QNM and ScanAsyst<sup>TM</sup>, is a new, Bruker-proprietary, primary Atomic Force Microscopy (AFM) mode. Other primary AFM modes include Contact, Tapping, Scanning Tunnelling Microscopy (STM) and Torsional Resonance modes.

Peak Force Tapping mode oscillates, but far below the cantilever resonant frequency, the vertical motion of the cantilever using the (main) Z piezo element and relies on peak force for feedback. Peak interaction force and nanoscale material property information is collected for each individual tap.

Because Peak Force Tapping mode does not resonate the cantilever, cantilever tuning is not required. This is particularly advantageous in fluids.

Peak Force Tapping Mode includes auto-optimization (called ScanAsyst) of scanning parameters, including gains, setpoint and scan rate. This enables users to rapidly obtain high quality images. ScanAsyst is intended to be the first choice imaging mode for NanoScope version 8.10 and later software.

Because Peak Force Tapping mode controls the applied force, tip wear is reduced.

Peak Force Tapping mode imaging increases the resolution by controlling the force that the tip applies to the sample thereby decreasing the deformation depths; this decreases the contact area

between the tip and sample. Because the deformation depths and lateral forces are small, there is minimal damage to the probe or sample.

## 1.2 What is in the PeakForce QNM kit?

The PeakForce QNM kit consists of the following items:

1. Software keys to enable PeakForce QNM in real-time and off-line operation.
2. A pack of ten each of the following probes:
  - ScanAsyst-Air
  - Tap150A, P/N MPP-12120-10
  - Tap300A (RTESPA), P/N MPP-11120-10
  - Tap525A, P/N MPP-13120-10
3. One pre-mounted DNISP-HS probe in the appropriate probe holder.
4. PeakForce QNM samples. Refer to **PeakForce QNM Samples**: [Chapter 3](#) for details.
5. One day of PeakForce QNM applications training

## 1.3 Conventions and Definitions

In the interest of clarity, certain nomenclature is preferred. An SPM *probe* is comprised of a *tip* affixed to a *cantilever* mounted on a *substrate*, which is inserted in a *probe holder*.

Three font styles distinguish among contexts. For example:

Window or Menu Item / **BUTTON OR PARAMETER NAME** is set to VALUE.

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# Chapter 2    PeakForce QNM Operation

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## 2.1    Introduction

This chapter describes how to perform a simple PeakForce QNM experiment. Later sections will discuss PeakForce QNM parameters and their influence on the measurements.

## 2.2    PeakForce QNM Principles of Operation

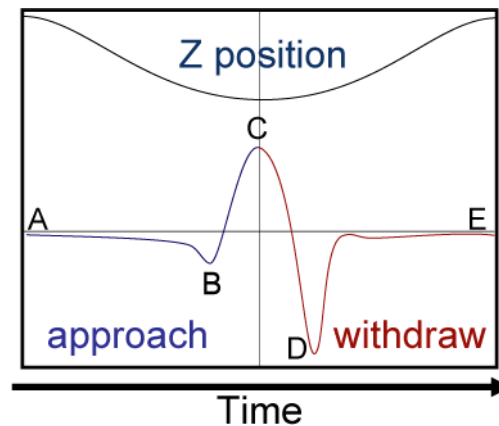
### 2.2.1    Peak Force Tapping Mode

Peak Force Tapping mode, the core technology behind PeakForce QNM and ScanAsyst modes, performs a very fast force curve at every pixel in the image. The peak interaction force of each of these force curves is then used as the imaging feedback signal. Peak Force Tapping mode modulates the Z-piezo at ~2 kHz (Icon, MultiMode. Catalyst operates at ~1 kHz) with a default **Peak Force Amplitude** of 150 nm (0-peak). Analysis of force curve data is done on the fly, providing a map of multiple mechanical properties that has the same resolution as the height image.

## 2.2.2 The “Heartbeat”

The **Force vs. Time** display, shown in [Figure 2.2a](#) is referred to as the “heartbeat.” The initial contact of the probe with the sample (B), peak force (C) and adhesion (D) points are labelled.

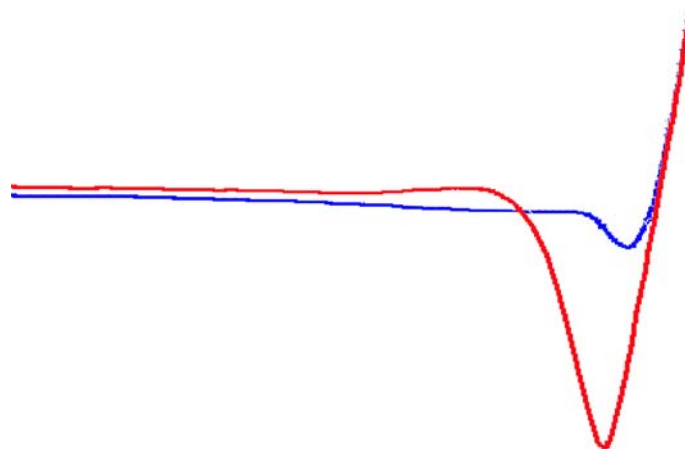
**Figure 2.2a** The “heartbeat.” Blue indicates approach while red indicates retract.



## 2.2.3 Force curves

Using the Z-position information, the heartbeat is transformed into a force curve, shown in [Figure 2.2b](#). The force curve plot is analyzed, on the fly, to produce the peak interaction force as the control feedback signal and the mechanical properties of the sample (**Adhesion**, **Modulus**, **Deformation**, **Dissipation**).

**Figure 2.2b** Force curve



## 2.3 PeakForce QNM Probe Selection

It is important to choose a probe that can cause enough deformation of the sample and still retain high force sensitivity. Therefore cantilever stiffness should be selected based on the sample stiffness. Bruker's recommendations are shown in Table 2.3a.

**Table 2.3a** Recommended Probes

Sample Modulus (E)	Probe	Nominal Spring Constant (k)
1 MPa < E < 20 MPa	ScanAsyst-Air	0.5 N/m
5 MPa < E < 500 MPa	Tap150A, P/N MPP-12120-10	5 N/m
200 MPa < E < 2000 MPa	Tap300A (RTESPA), P/N MPP-11120-10	40 N/m
1 GPa < E < 20 GPa	Tap525A, P/N MPP-13120-10	200 N/m
10 GPa < E < 100 GPa	DNISP-HS	350 N/m

**Note:** The recommended spring constants are general guidelines that reflect a compromise between image resolution and modulus accuracy. E.g. a stiff cantilever will improve modulus accuracy at the expense of damaging the sample.

To reduce optical interference, probes should be coated on their back side.

You may purchase these probes from Bruker Probes, <http://www.brukerafmprobes.com>.

## 2.4 Basic PeakForce QNM Operation

This section describes how to perform a simple PeakForce QNM experiment. Later sections will discuss PeakForce QNM parameters and their influence on the measurements.

### 2.4.1 Select the Microscope

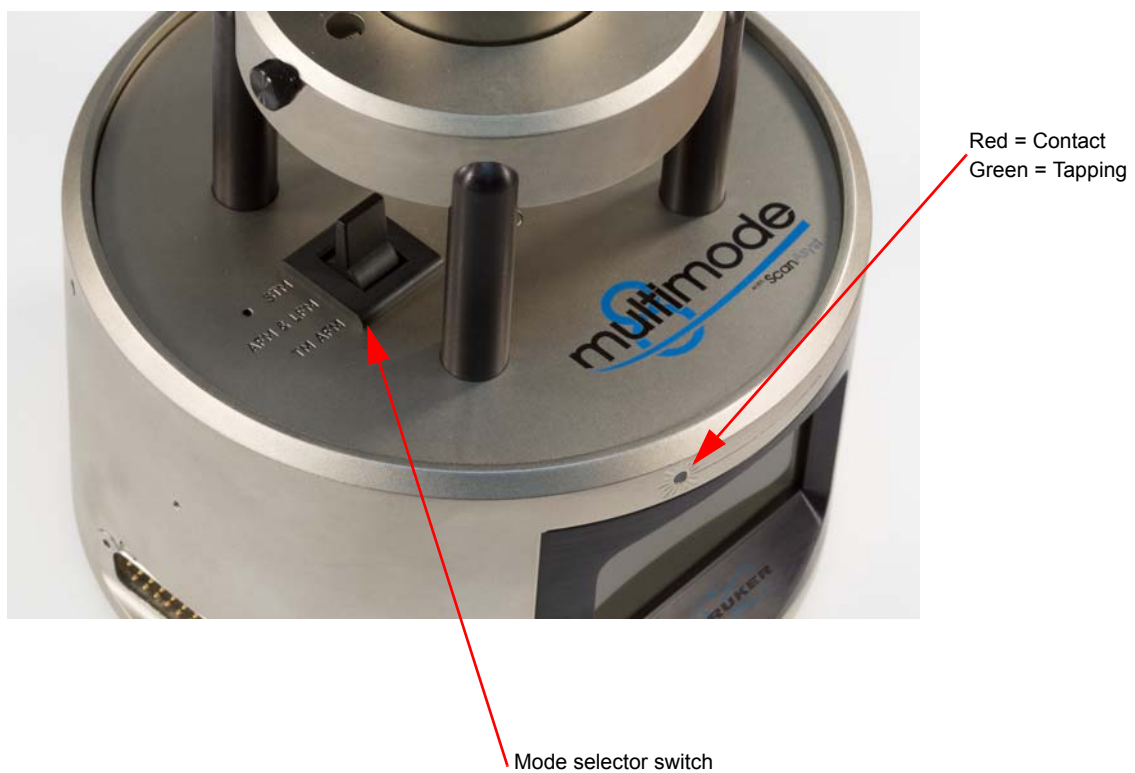
Follow the Select Microscope procedure described in your microscope Instruction Manual.



## 2.4.2 Configure the Hardware

1. If you have a MultiMode, set the mode selector switch on the MultiMode base to **AFM & LFM**.  
See [Figure 2.4a](#).

**Figure 2.4a** Mode selector switch

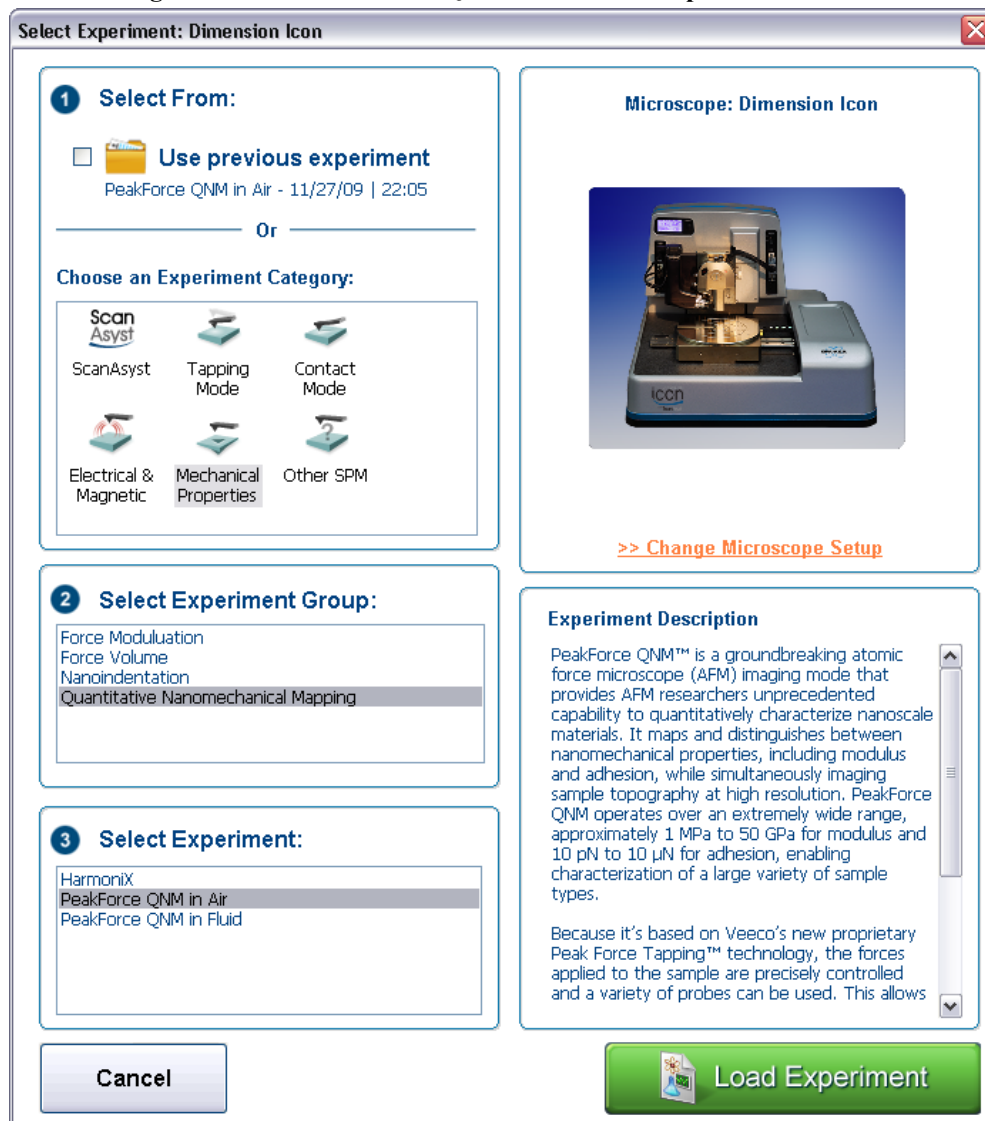


## 2.4.3 Select Mode of Operation



1. Click the **SELECT EXPERIMENT** icon. This opens the **Select Experiment** window, shown in Figure 2.4b.

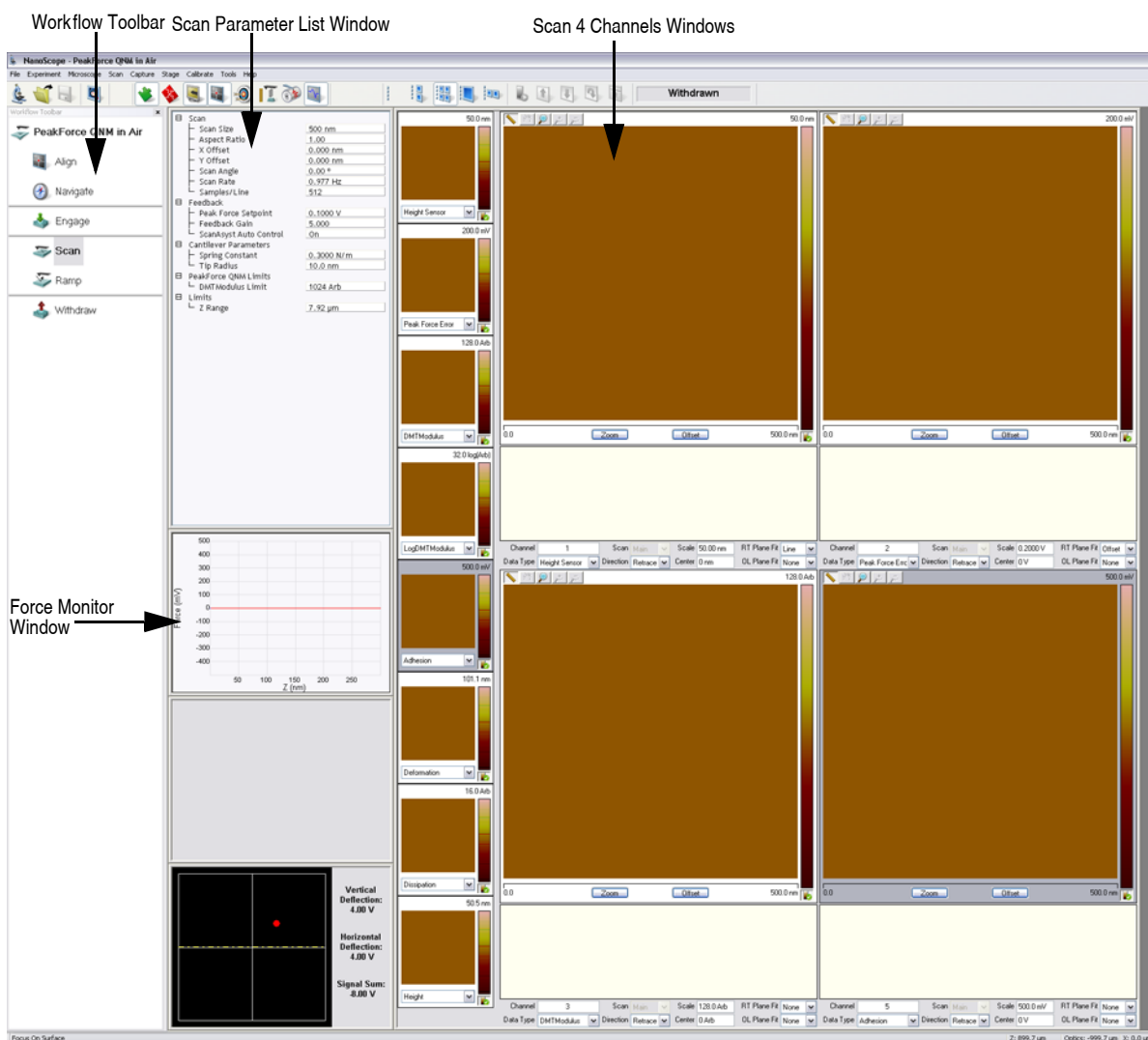
Figure 2.4b The PeakForce QNM in Air **Select Experiment** window



2. Select **MECHANICAL PROPERTIES** in the **Experiment Category** panel.
3. Select **QUANTITATIVE NANOMECHANICAL MAPPING** in the **Select Experiment Group** panel.
4. Select **PEAKFORCE QNM IN AIR** in the **Select Experiment** panel and click **LOAD EXPERIMENT**.

5. This opens the **Workflow Toolbar**, the **Scan 4 Channels** (Icon) windows, the **Force Monitor** window and the **Scan Parameters List** window, shown in [Figure 2.4c](#).

**Figure 2.4c** PeakForce QNM in Air (Simple Mode) configuration



## 2.4.4 Head, Cantilever and Sample Preparation

1. Install a suitable probe onto an AFM cantilever holder. See **PeakForce QNM Probe Selection**: [Section 2.3](#).
2. Load the cantilever holder with installed tip into your microscope.

### 2.4.5 Align Laser

1. Align the laser using the laser control knobs.

**Note:** Coated cantilevers are strongly recommended to increase the laser sum signal and decrease interference effects.

**Note:** Maximize the laser sum signal to avoid optical interference.

**Note:** Try not to change the laser spot position during the experiment. This may change the **Deflection Sensitivity** and therefore the property measurements.

### 2.4.6 Adjust Photodetector

1. Adjust the photodetector.

## 2.4.7 Set Initial Scan Parameters

### Scan Panel

In the **Scan** panel of the **Scan Parameters List**, set the following initial scan parameters (see [Figure 2.4d](#)).

1. Set the **Scan Size**.
2. Set the **Scan Angle**.

### Feedback Panel

1. Set **ScanAsyst Auto Control** to **ON** (see [Figure 2.4d](#)).

**Figure 2.4d PeakForce QNM in Air (SIMPLE MODE) Parameters Panel**

Scan	
Scan Size	500 nm
Aspect Ratio	1.00
X Offset	0.000 nm
Y Offset	0.000 nm
Scan Angle	0.00 °
Scan Rate	0.977 Hz
Samples/Line	512
Feedback	
Peak Force Setpoint	0.1000 V
Feedback Gain	5.000
ScanAsyst Auto Control	On
Cantilever Parameters	
Spring Constant	0.3000 N/m
Tip Radius	10.0 nm
Sample Poisson's Ratio	0.300
PeakForce QNM Limits	
DMT Modulus Limit	1024 Arb
Limits	
Z Range	7.42 µm
Other	
Units	Metric

## Channels

1. Set the **Channel 1 Data Type** to **HEIGHT SENSOR** (see [Figure 2.4e](#)).
2. Set the **Channel 2 Data Type** to **PEAK FORCE ERROR** (see [Figure 2.4e](#)).
3. Set the **Channel 3 Data Type** to **DMT MODULUS** (see [Figure 2.4e](#)).
4. Set the **Channel 4 Data Type** to **LOGDMT MODULUS** (see [Figure 2.4e](#)).
5. Set the **Channel 5 Data Type** to **ADHESION** (see [Figure 2.4e](#)).
6. Set the **Channel 6 Data Type** to **DEFORMATION** (see [Figure 2.4e](#)).
7. Set the **Channel 7 Data Type** to **DISSIPATION** (see [Figure 2.4e](#)).
8. Set **Data Scale** to a reasonable value for the sample or click the **AUTOSCALE** icon after engaging.



**Note:** For example, for a 200nm step height calibration sample, a reasonable **Data Scale** setting is 300nm initially.

9. Set **Line direction** to either **TRACE** or **RETRACE**.

**Figure 2.4e** Suggested PeakForce QNM Channel Settings

Channel	1	Scan	Main	Scale	50.00 nm	RT Plane Fit	Line
Data Type	Height Sensor	Direction	Retrace	Center	0 nm	OL Plane Fit	None
Channel	2	Scan	Main	Scale	0.2000 V	RT Plane Fit	Offset
Data Type	Peak Force Error	Direction	Retrace	Center	0 V	OL Plane Fit	None
Channel	3	Scan	Main	Scale	128.0 Arb	RT Plane Fit	None
Data Type	DMTModulus	Direction	Retrace	Center	0 Arb	OL Plane Fit	None
Channel	4	Scan	Main	Scale	32.00 log(Arb)	RT Plane Fit	None
Data Type	LogDMTModulus	Direction	Retrace	Center	0 log(Arb)	OL Plane Fit	None
Channel	5	Scan	Main	Scale	500.0 mV	RT Plane Fit	None
Data Type	Adhesion	Direction	Retrace	Center	0 V	OL Plane Fit	None
Channel	6	Scan	Main	Scale	101.1 nm	RT Plane Fit	None
Data Type	Deformation	Direction	Retrace	Center	0 nm	OL Plane Fit	None
Channel	7	Scan	Main	Scale	16.00 Arb	RT Plane Fit	None
Data Type	Dissipation	Direction	Retrace	Center	0 Arb	OL Plane Fit	None



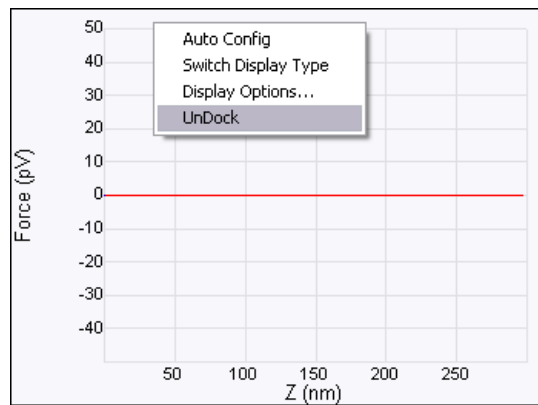
## 2.4.8 Engage

1. Select **Microscope > Engage** or click the **ENGAGE** icon on the Workflow Toolbar. A pre-engage check begins, followed by Z-stage motor motion.
2. To move to another area of the sample, execute a **Withdraw** command to avoid damaging the tip and scanner.

## 2.4.9 Image the sample

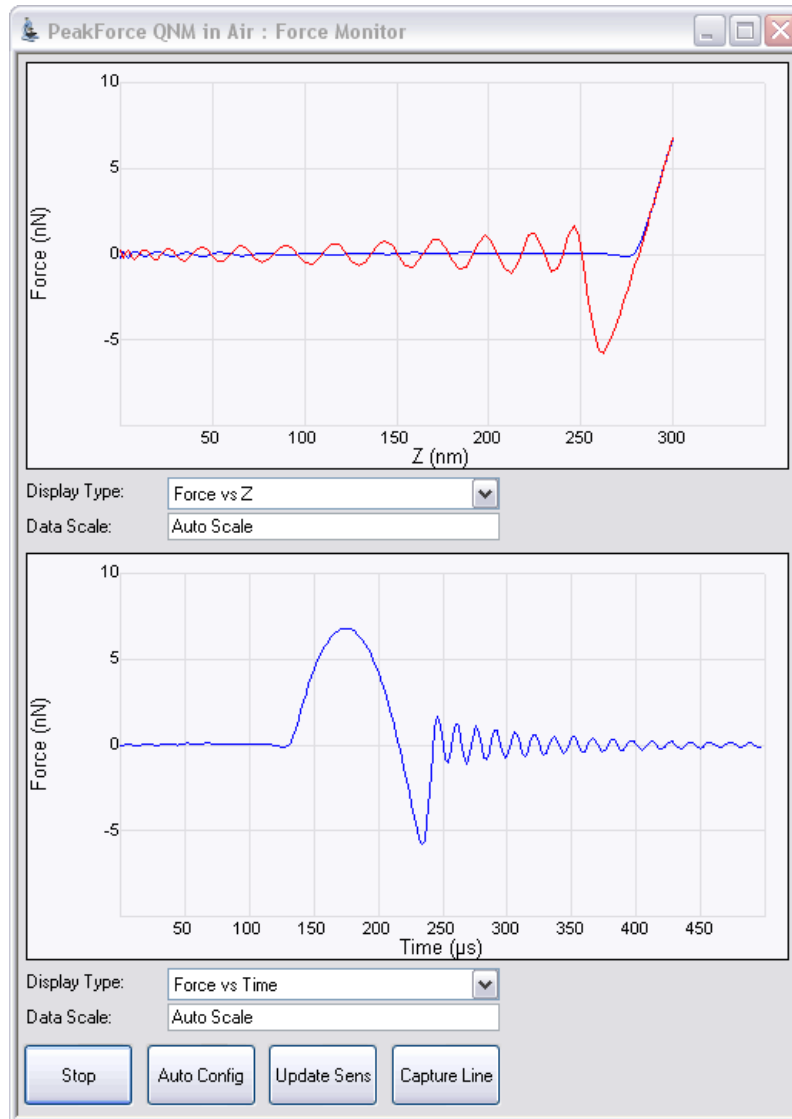
1. If needed, right-click in the **Force Monitor** window and click **UNDOCK**. See [Figure 2.4f](#). You may **DOCK** the undocked **Force Monitor** window by right-clicking in it and clicking **DOCK**.

**Figure 2.4f** Undock the Force Monitor window



2. Select one plot to be **FORCE vs. TIME** and the other to be **FORCE vs. Z**.
3. Once scanning, the **Force Monitor** window, shown in [Figure 2.4g](#), should display a **Force vs. Z** plot and a “heartbeat” (**Force vs. Time**) plot.

**Figure 2.4g Force Monitor window**

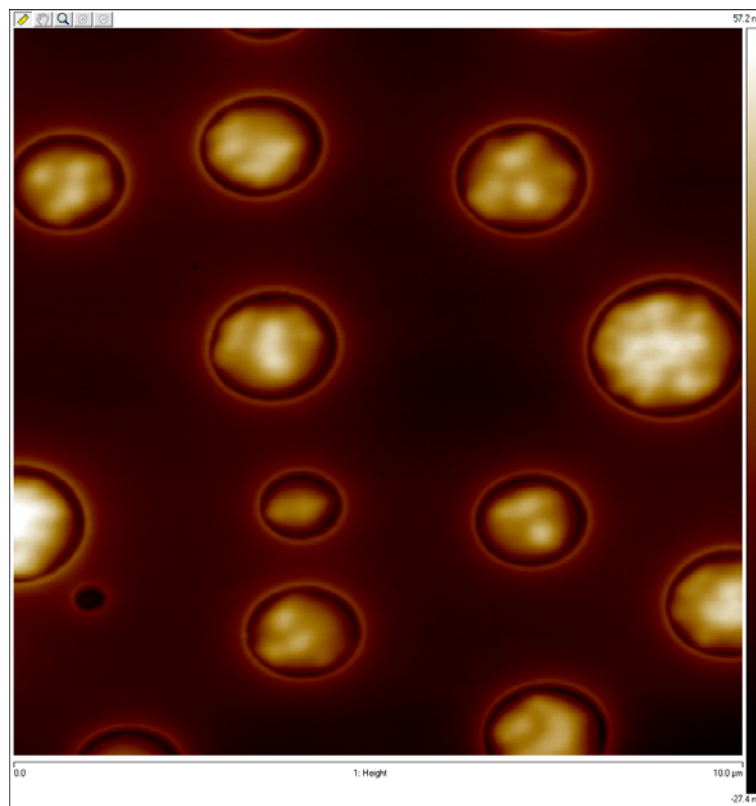


**Note:** The cantilever oscillation after it snaps off the sample surface, shown in [Figure 2.4g](#), is normal. On occasion this oscillation will continue until the probe tip again contacts the sample surface. This oscillation will be heavily damped by this contact. Even if the oscillation is not fully damped, the remaining oscillation at the force peak will be small and will merely add a small amount of noise to the feedback.



4. The **HEIGHT** channel in the **Scan** window, shown in [Figure 2.4h](#), will display a topographical image of your sample.

**Figure 2.4h** Height Image of a PS + LPDE blend.



## 2.5 PeakForce QNM Channels

This section discusses channels that are specific to PeakForce QNM mode. Mechanical properties can be extracted from the calibrated (see [Chapter 4](#)) force curves.

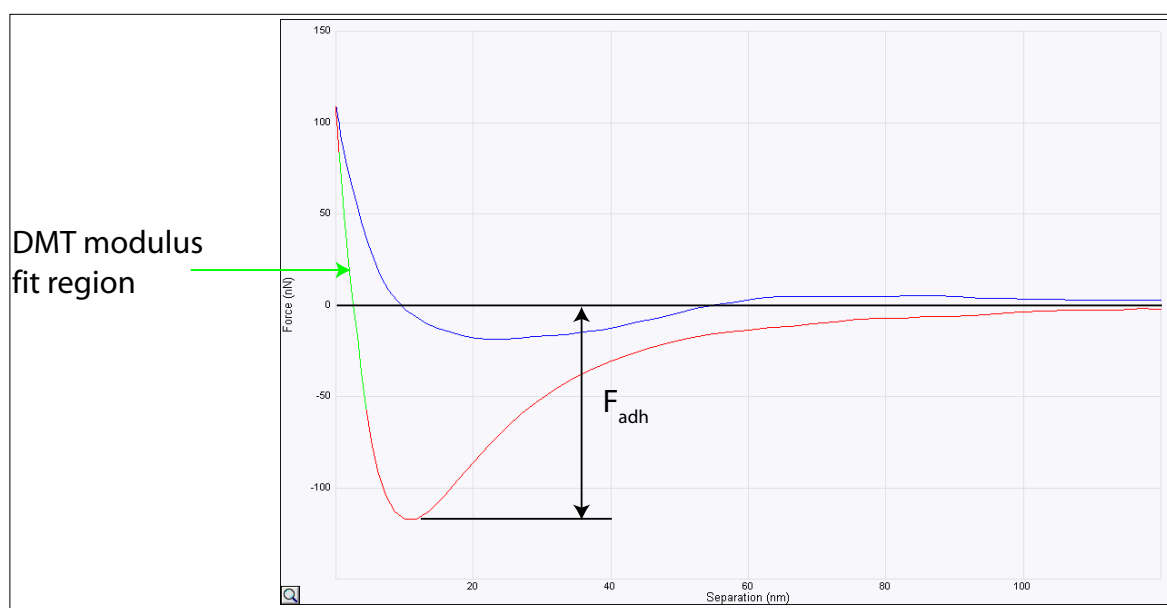
### 2.5.1 DMT Modulus

The reduced Young's Modulus,  $E^*$ , is obtained by fitting the retract curve (green line in [Figure 2.5a](#)) using the Derjaguin, Muller, Toropov (DMT) model<sup>1</sup> given by

$$F_{tip} = \frac{4}{3}E^*\sqrt{Rd^3} + F_{adh}$$

Where  $F_{tip}$  is the force on the tip,  $F_{adh}$  is the adhesion force,  $R$  is the tip end radius and  $d$  is the tip-sample separation.

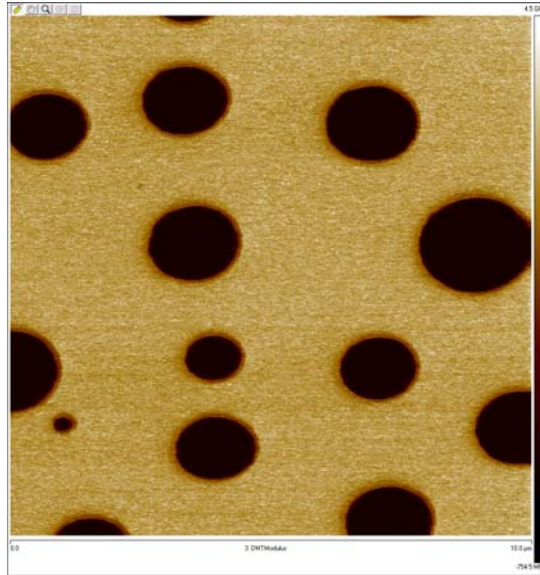
**Figure 2.5a** Force vs. Separation plot



1. Derjaguin B.V., Muller V.M., Toropov Yu.P., J. Colloid. Interface Sci. 53, 314 (1975).

Figure 2.5b shows a DMT Modulus map of PS+LDPE blend.

**Figure 2.5b** DMT Modulus map of a PS+LDPE blend



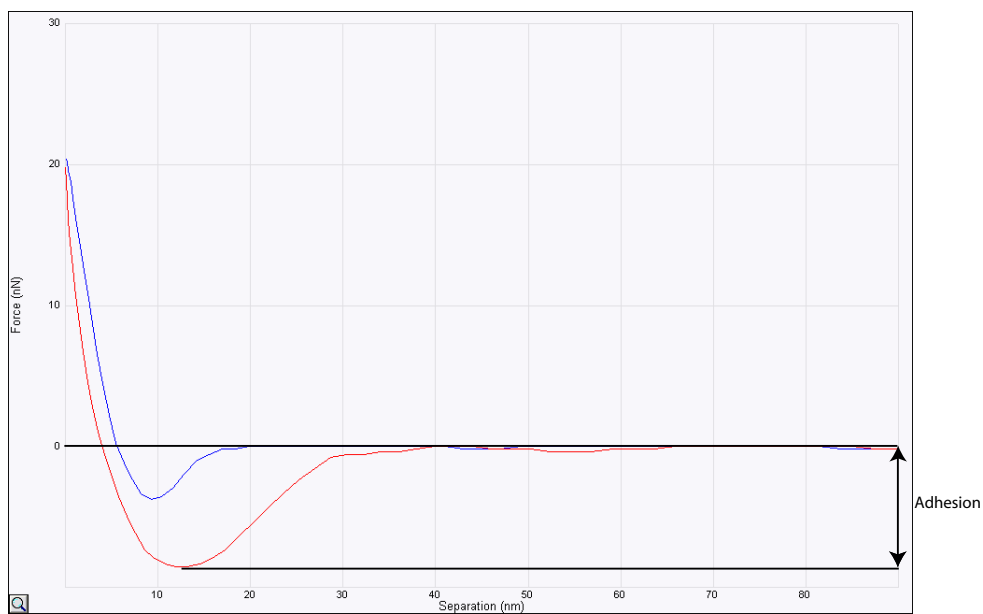
## 2.5.2 Log DMT Modulus

The logarithm of the elastic modulus of the sample based on the DMT model.

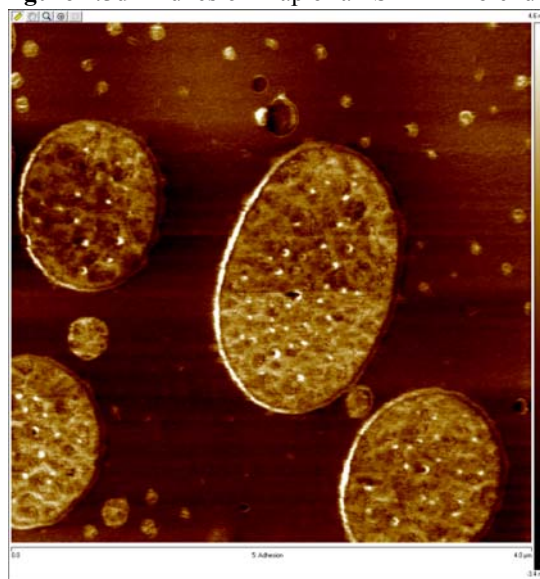
### 2.5.3 Adhesion

The peak force below the baseline, shown in [Figure 2.5c](#). [Figure 2.5d](#) shows an adhesion map of a PS+LDPE blend.

**Figure 2.5c** Adhesion on a PS+LDPE blend



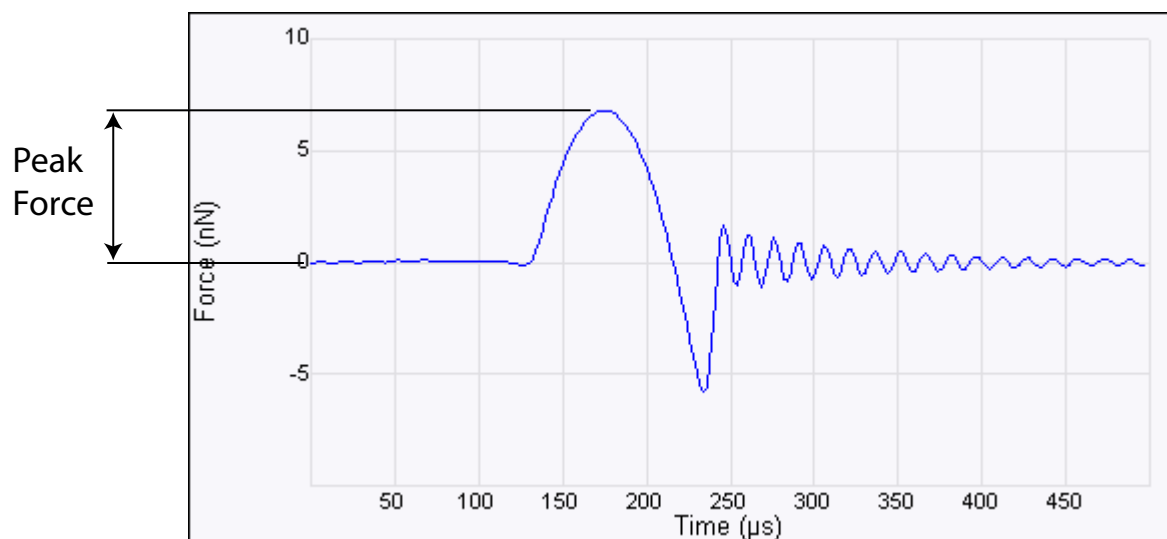
**Figure 2.5d** Adhesion map of a PS+LDPE blend



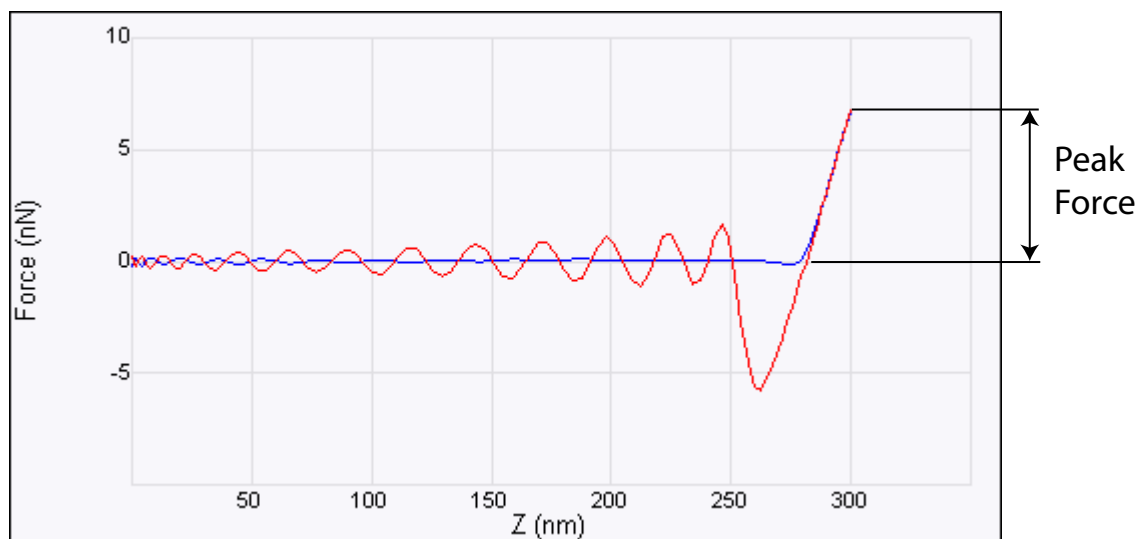
## 2.5.4 Peak Force

This channel produces a map of the peak force (see [Figure 2.5g](#)) measured during the scan. Because the PeakForce QNM mode uses peak force as the feedback signal, this channel is essentially the **Peak Force Setpoint** plus the error. [Figure 2.5e](#) and [Figure 2.5f](#) illustrate the peak force location.

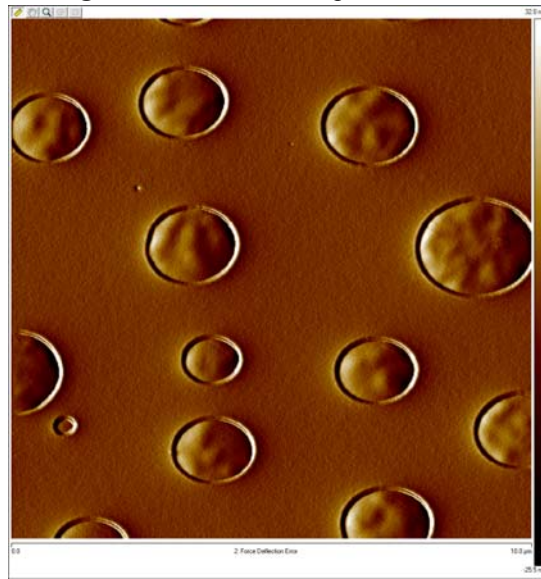
**Figure 2.5e** The “heartbeat,” Force vs. Time



**Figure 2.5f** Force curve, Force vs. distance



**Figure 2.5g** Peak Force Error map of a PS+LDPE blend



## 2.5.5 Dissipation

Energy Dissipation ( $W$ ) is given by the force times the velocity integrated over one period of the vibration:

$$W = \int_0^T \vec{F} \cdot \vec{v} dt = \int \vec{F} \cdot d\vec{Z}$$

$F$  is the interaction force vector and  $dZ$  is the displacement vector. Because the  $Z$  motion and the velocity reverse direction in a half cycle, the integral is zero if the load and unload curves coincide. The dissipation is therefore the hysteresis between the load and unload curves. Pure elastic deformation has no hysteresis which corresponds to very low dissipation. Energy dissipated is displayed in electron volts as the mechanical energy lost per tapping cycle.

The **Dissipation** channel plots the dissipated energy in each cycle by integrating the area between the Trace (load or extend) and Retrace (unload or retract) curves as shown in the blue area in [Figure 2.5h](#).

**Figure 2.5h** Dissipation (shaded area) in a polystyrene (PS) and Low-density polyethylene (LPDE) blend

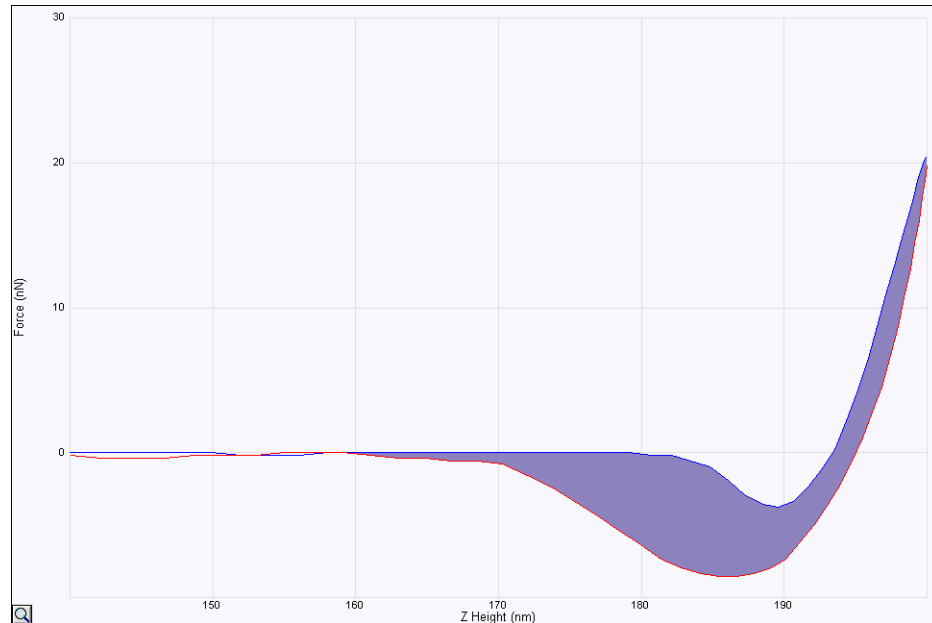
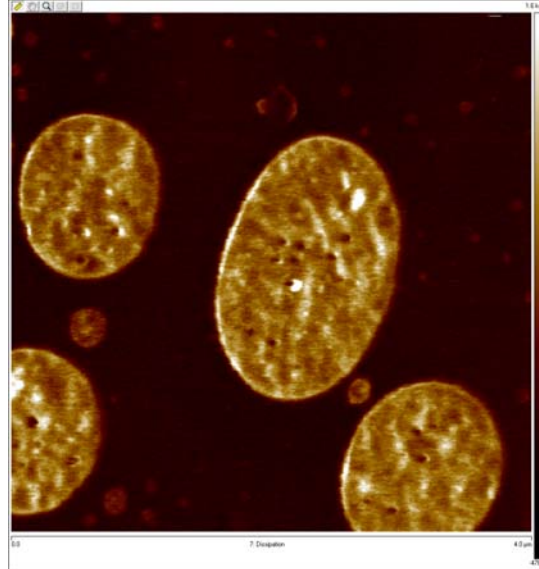


Figure 2.5i shows the dissipation image of a PS+LDPE blend.

**Figure 2.5i** Dissipation image of a PS+LDPE blend



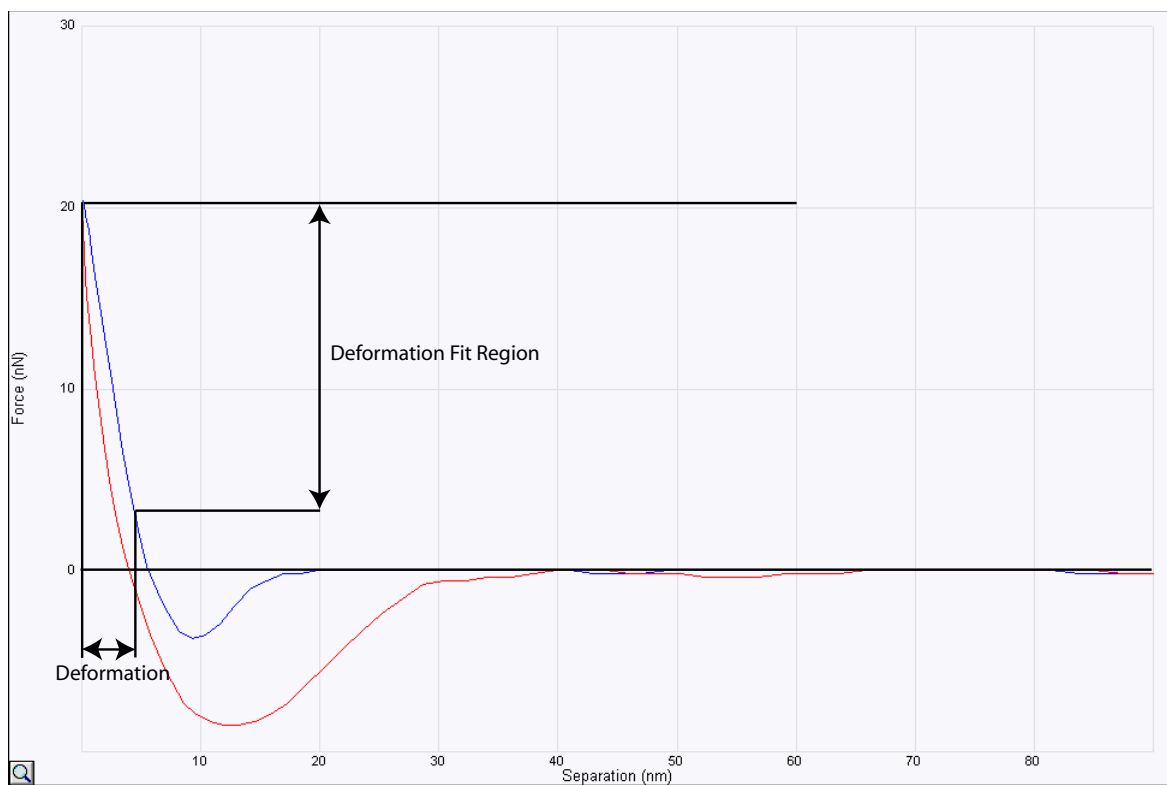


## 2.5.6 Deformation

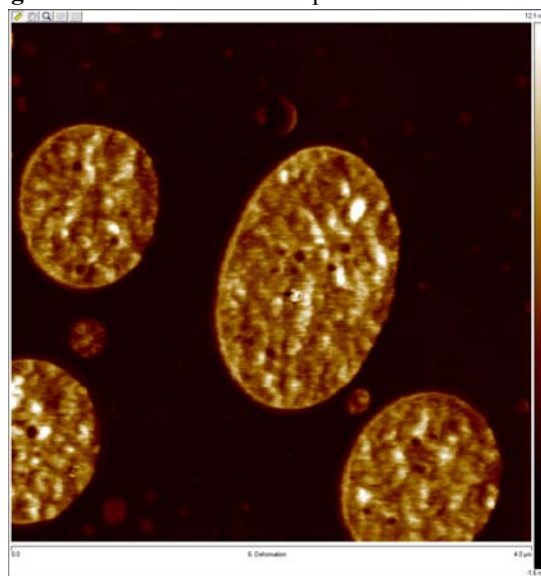
The maximum deformation of the sample (defined as the distance from the base of the **Deformation Fit Region** position to the peak interaction force position) caused by the probe. See [Figure 2.5j](#). [Figure 2.5k](#) shows a deformation map of a PS+LPDE blend.

**Note:** The total deformation will be slightly larger than the displayed deformation because the default **Deformation Fit Region** is 85% of the full deformation. See **Deformation Fit Region:** [Page 28](#).

Figure 2.5j Deformation



**Figure 2.5k** Deformation map of a PS+LDPE blend



## 2.6 PeakForce QNM Parameters

### 2.6.1 Feedback Parameters

#### Peak Force Setpoint

The setpoint for peak force. If the deflection sensitivity is calibrated, the force (in Newtons) will be displayed.

When the **ScanAsyst Setup** is **ON**, **Peak Force Setpoint** is automatically controlled by NanoScope software. Under some conditions, you may desire to control the **Peak Force Setpoint** manually. A **Peak Force Setpoint** that is too high can either damage the sample or wear the tip. It is generally desirable to reduce the **Peak Force Setpoint** to as small a value as is possible. However, in order to achieve accurate Elastic modulus measurement, sufficient sample deformation is needed. If the deformation is less than 2nm, increase the **Peak Force Setpoint** to achieve sufficient sample deformation.

**Note:** When performing **AUTO CONFIG** operations with a small **Peak Force Setpoint** (less than ~20mV), the tip may drift out of contact with the surface and will be unable to return and track the surface. It is therefore recommended using a relatively large **Peak Force Setpoint** while performing **AUTO CONFIG** operations and reducing the **Peak Force Setpoint** later if necessary.

### Feedback Gain

The gain of the Peak Force Tapping feedback control loop.

**Note:** Both **Peak Force Setpoint** and **Feedback Gain** are dynamically and automatically controlled when **ScanAsyst Auto Control** is set to **ON**.

**Note:** A **Feedback Gain** that is too large will cause oscillation of the system and increase noise, while too small a **Feedback Gain** will result in poor sample tracking.

### Low Pass Deflection Bandwidth

The low pass filter is used to reduce deflection noise. Lower bandwidths will reduce noise but will distort the force curve and introduce errors in quantitative nanomechanical property measurements.

*Range and Settings:* 10 kHz - 65.56 kHz (Default value: 40 kHz).

### ScanAsyst Setup

*Range and Settings:* **NEVER:** Does not allow ScanAsyst Auto Control.

**ALLOW:** Allows ScanAsyst Auto Control.

**Note:** **SHOW ALL**, discussed in the *NanoScope Software Version 8 User Guide*, must be enabled to view and edit this parameter

### ScanAsyst Noise Threshold

**ScanAsyst Noise Threshold** is linked to the **Feedback Gain** and is used to tune it. Larger **ScanAsyst Noise Thresholds** will result in better sample tracking but increased oscillation noise. Lower **ScanAsyst Noise Thresholds** will result in a cleaner image but the sample tracking will suffer.

*Range and Settings:* 0.5 nm is appropriate for most samples while 1 nm is appropriate for rough samples and 0.05 nm may be appropriate for very flat samples.

**Note:** When **ScanAsyst Auto Z Limit** control is turned **ON**, the **ScanAsyst Noise Threshold** parameter is automatically set by the program and cannot be changed.

### ScanAsyst Auto Config Frames

At the end of every N frames, an **AUTO CONFIG** operation is performed.

*Range and Settings:* 0 - 100. If **ScanAsyst Auto Config Frames** = 0, periodic **AUTO CONFIG** operations are not performed.

### ScanAsyst Auto Control

*Range and Settings:* **OFF**: Turns ScanAsyst Auto Control **OFF**.

**ON**: Turns ScanAsyst Auto Control **ON**.

**INDIVIDUAL**: Allows individual control of **ScanAsyst Auto Gain**, **ScanAsyst Auto Setpoint**, **ScanAsyst Auto Scan Rate** and **ScanAsyst Auto Z Limit**.

### ScanAsyst Auto Gain

**ScanAsyst Auto Gain** allows NanoScope to dynamically control **Feedback Gain**.

*Range and Settings:* **OFF**: Turns **ScanAsyst Auto Gain** **OFF**.

**ON**: Turns **ScanAsyst Auto Gain** **ON**.

### ScanAsyst Auto Setpoint

**ScanAsyst Auto Setpoint** allows NanoScope to dynamically control the **Peak Force Setpoint**.

*Range and Settings:* **OFF**: Turns **ScanAsyst Auto Setpoint** **OFF**.

**ON**: Turns **ScanAsyst Auto Setpoint** **ON**.

**Note:** This option is very useful for users who want to change the **Peak Force Setpoint** manually to achieve adequate deformation on the sample while leaving **ScanAsyst Auto Gain** **ON**.

### ScanAsyst Scan Auto Scan Rate

**ScanAsyst Scan Auto Scan Rate** allows NanoScope to control the **Scan Rate**.

*Range and Settings:* **OFF**: Turns **ScanAsyst Scan Auto Scan Rate** **OFF**.

**ON**: Turns **ScanAsyst Scan Auto Scan Rate** **ON**.

### ScanAsyst Auto Z Limit

**ScanAsyst Auto Z Limit** allows NanoScope to control the **Z Limit**. The **ScanAsyst Auto Z Limit** function will detect if the surface is sufficiently smooth to allow reduction of the **Z Limit** and thus avoid bit noise in the **Height** and **Height Sensor** channel. This will be effective after a whole frame of the image is scanned. If the **Z Limit** needs to be reduced, the **ScanAsyst Noise Threshold** will automatically be reduced to 0.15 times the original **ScanAsyst Noise Threshold** to reduce the oscillation noise for smooth samples.

*Range and Settings:* **OFF:** Turns **ScanAsyst Auto Z Limit** OFF.

**ON:** Turns **ScanAsyst Auto Z Limit** ON.

## 2.6.2 PeakForce QNM Control Parameters

### Peak Force Amplitude

The zero-to-peak amplitude of the cantilever drive in the Z axis (Z modulation). Increasing **Peak Force Amplitude** will reduce the contact time during each tip tapping cycle on the sample and help tracking the rough and/or sticky sample by avoiding a situation where the tip is unable to pull off from the sample. Reduced **Peak Force Amplitude** is desired in liquid on flat samples. Less **Peak Force Amplitude** results in less hydrodynamic force disturbance.

### Lift Height

The distance that the Z-piezo is retracted from the sample during an **AUTO CONFIG** operation.

**Note:** Changing the **Lift Height** will automatically start the **AUTO CONFIG** function (see **Optimizing a ScanAsyst Image:** [Page 38](#)) and retract the Z piezo to the specified **Lift Height**. Clicking **AUTO CONFIG** will automatically calculate the **Lift Height** and perform an **AUTO CONFIG** operation.

## Top Fit Region

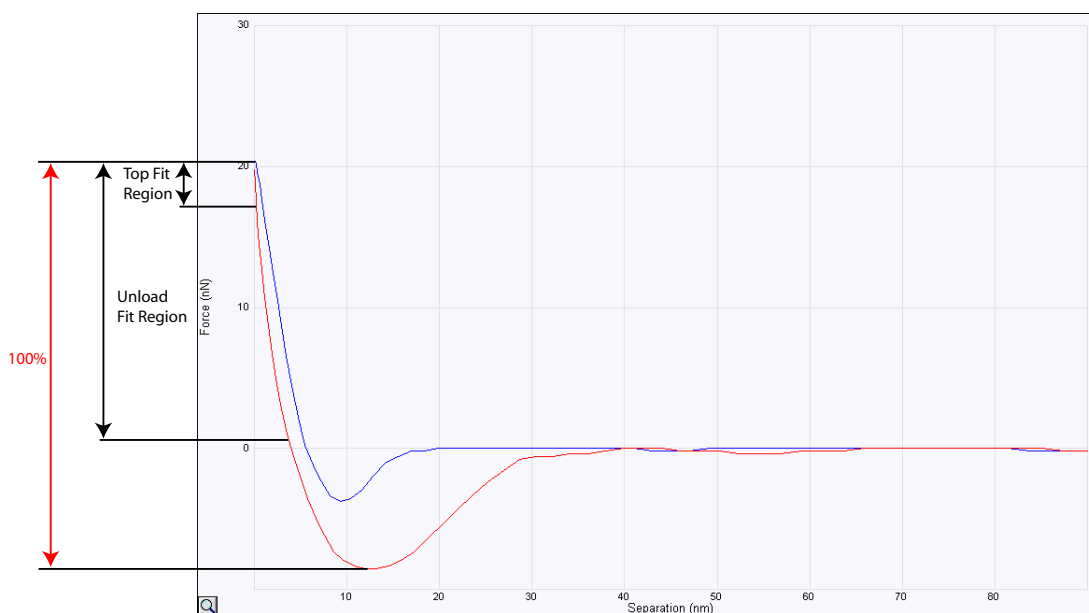
The **Top Fit Region** of the unload force curve, shown in [Figure 2.6a](#), is excluded from the DMT Modulus calculations.

**Note:** A smaller **Top Fit Region** means that less region of the force curve is excluded from the DMT modulus calculations.

*Range and Settings:* 0 - 94%.

*Typical:* 10%.

**Figure 2.6a** DMT Fit regions of the Force curve



## Unload Fit Region

The **Unload Fit Region** of the force curve, shown in [Figure 2.6a](#), is included in the DMT Modulus calculations.

*Range and Settings:* 0 - 100%. 100% is defined as the force between the adhesion point and the peak force.

*Typical:* 70%.

The portion of the force curve between the **Top Fit Region** and the **Unload Fit Region** is included in the DMT Modulus calculations. For typical numbers discussed here, the region between 10% and 70% of the unload force curve will be included in the DMT modulus calculations.

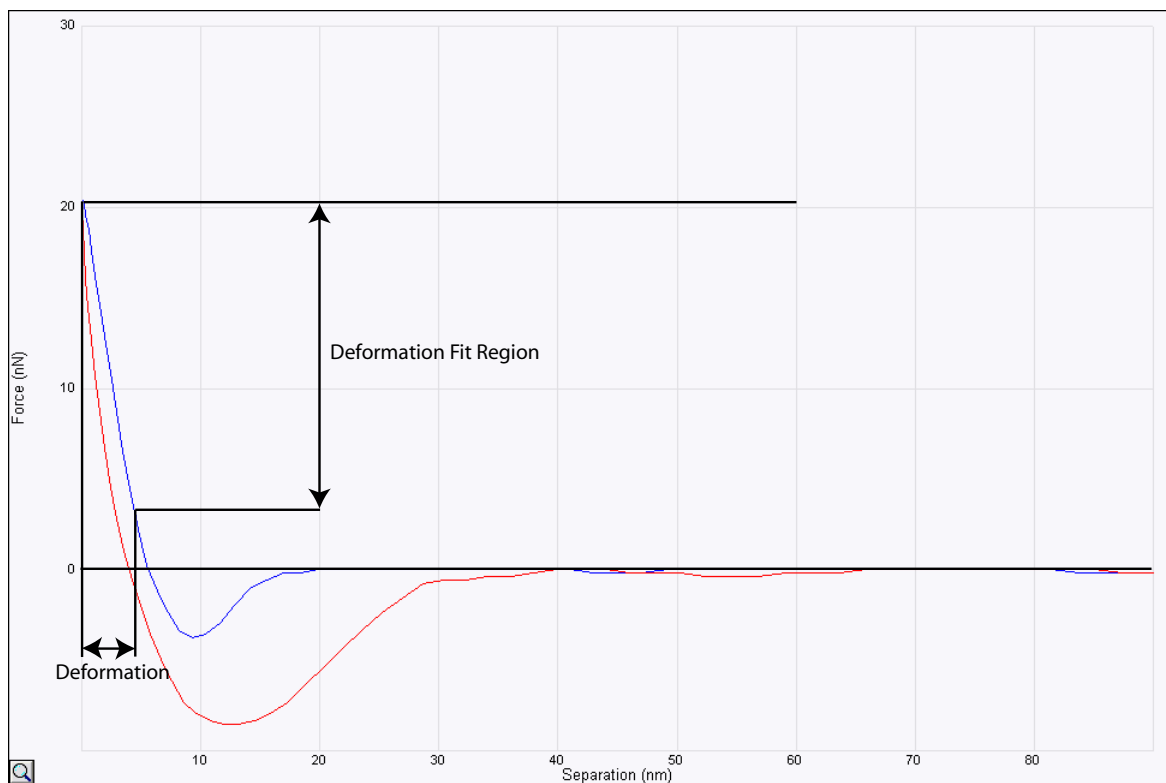
### Deformation Fit Region

The **Deformation Fit Region** of the load force curve, shown in [Figure 2.6b](#), is excluded from the **Deformation** channel display. This parameter is used to reduce the effect of baseline noise.

*Range and Settings: 50 - 100%.* 100% is defined as the force between the zero force point and the peak force in the load curve.

*Typical: 85%.* The portion of the force curve above the 85% point is displayed in the Deformation channel.

**Figure 2.6b** DMT Fit regions of the Force curve



### 2.6.3 Cantilever Parameters

The following parameters are needed to calibrate PeakForce QNM.

#### Spring Constant

Measure the spring constant of the probe and input that value into this panel. Spring constant may be measured using the Thermal Tune function in NanoScope software. Refer to **Calibrate the Spring Constant Using Thermal Tuning**: [Section 4.4](#) for details.

#### Tip Radius

Measure the tip radius and input the value in this panel. Tip radius may be measured using a tip characterizer sample and the Tip Qualification function in NanoScope software. Refer to **Measure the Tip Radius**: [Section 4.5](#) for details.

#### Poisson's Ratio

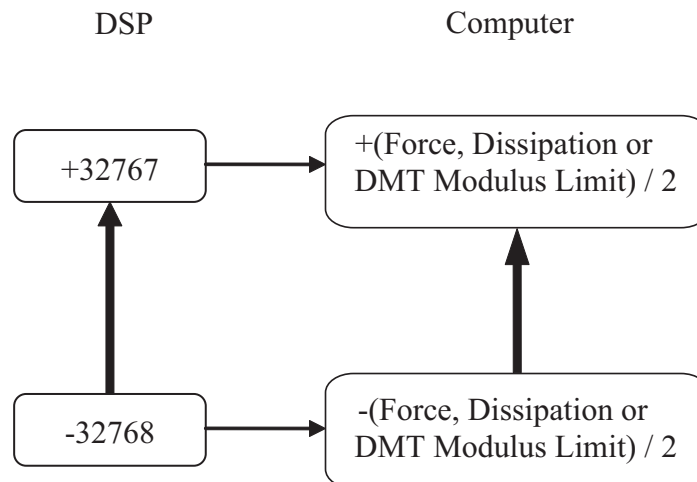
Poisson's ratio of the sample. This is used to calculate the sample modulus,  $E_s$ , from the measured reduced modulus,  $E^*$ : Refer to **Calibrate Peak Force QNM**: [Section 4.6](#) for details.



## 2.6.4 PeakForce QNM Limits

These parameters work like other limits in the NanoScope software. Numbers in the NanoScope software are represented using 16 bits and thus various quantities are represented as illustrated in [Figure 2.6c](#).

**Figure 2.6c** Illustration of **PeakForce QNM Limits**



As with other limits, setting the limit too high increases the bit noise. Setting the limit too low can result in wrapped data and inverted contrast. The limits of the following parameters can be set by the user:

- Force Limit: affects the **Peak Force** and **Adhesion** channels.
- Dissipation Limit: affects the **Dissipation** channel.
- DMT Modulus Limit: affects the **DMT Modulus** channel.
- LogDMT Modulus Limit: affects the **LogDMT Modulus** channel.
- LogDMT Modulus Offset: affects the **LogDMT Modulus** channel.

## 2.6.5 Limits Parameters

### Z Limit

Permits attenuation of maximum allowable Z voltage and vertical scan range to achieve higher resolution (smaller quantization) in the Z direction.

*Range or Settings:*

- Dimension Icon: 8.33 V (~0.241  $\mu\text{m}$ ) to 309.3 V (~9  $\mu\text{m}$ ).
- MultiMode 8: 11 V (~0.1375  $\mu\text{m}$ ) to 416 V (~5  $\mu\text{m}$ ).
- BioScope Catalyst: 1 V (~0.1252  $\mu\text{m}$ ) to 145 V (~18  $\mu\text{m}$ ).

**Note:** **SHOW ALL**, discussed in the *NanoScope Software Version 8 User Guide*, must be enabled to view and edit this parameter.

### Z Range

Permits attenuation of the range of the Z piezo as measured by the Z sensor to achieve higher resolution (smaller quantization) in the Z direction.

*Range or Settings:*

- Dimension Icon: 0.2 nm to ~10 microns
- BioScope Catalyst: 0.2 nm to ~28 microns

**Note:** The **Z Range** parameter does not apply to the MultiMode because it does not incorporate a Z sensor.

### Deflection Limit

Use this parameter to attenuate the maximum allowable deflection signal to achieve higher resolution. If this value is too small, saturation of the **Deflection** channel will occur.

*Range or Settings:* 4.096V - 24.58V.

## 2.6.6 Other Parameters

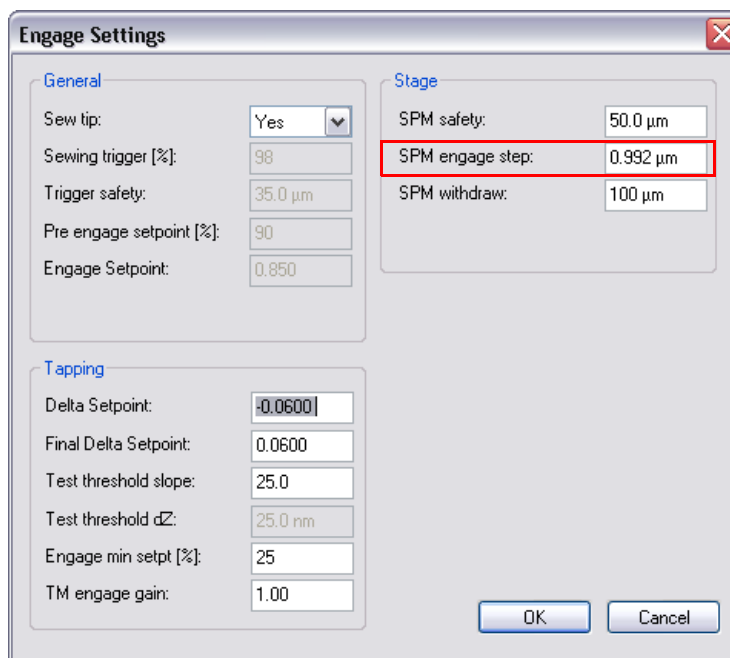
### Peak Force Engage Setpoint

Laser interference from reflective samples can cause a “false engage” which can be avoided by using a large **Peak Force Engage Setpoint**. This is why a relatively large and conservative **Peak Force Engage Setpoint** default value is used. But large **Peak Force Engage Setpoints** can damage samples and probes, particularly cantilevers with high spring constants. To reduce the engage force, reduce the **Peak Force Engage Setpoint**. When you reduce **Peak Force Engage Setpoint**, you should also reduce **SPM engage step**, found in **Microscope > Engage Settings > General**, shown in [Figure 2.6d](#).

*Range or Settings:*

- Dimension Icon: 0.001 V - 1.229 V (*Default value: 0.15 V*).
- MultiMode 8: 0.001 V - 1.229 V (*Default value: 0.15 V*).
- BioScope Catalyst: 0.001 V - 1.229 V (*Default value: 0.3 V*).

**Figure 2.6d** SPM engage step



## **Medium**

The medium surrounding the sample and probe. This parameter is selected when you select either PeakForce QNM in Air

*Range and Settings:* **AIR.**

**FLUID.**

**Note:** **SHOW ALL**, discussed in the *NanoScope Software Version 8 User Guide*, must be enabled to view and edit this parameter

## 2.6.7 Parameter Visibility

The visibility of various parameters depends on the selected mode. [Table 2.6a](#) shows parameter visibility as a function of microscope mode.

**Table 2.6a** Parameter Visibility

Panel	Parameter	Simple Mode	Expanded Mode	Show All	Other Dependencies
Feedback	Peak Force Setpoint	Yes	Yes	Yes	
	Feedback Gain	Yes	Yes	Yes	
	Low Pass Deflection Bandwidth	No	Yes	Yes	
	ScanAsyst Setup	No	No	Yes	
	ScanAsyst Noise Threshold	No	Yes	Yes	
	ScanAsyst Auto Config Frames	No	No	Yes	
	ScanAsyst Auto Control	Yes	Yes	Yes	
	ScanAsyst Auto Gain	Yes	Yes	Yes	ScanAsyst Auto Control
	ScanAsyst Auto Setpoint	Yes	Yes	Yes	ScanAsyst Auto Control
	ScanAsyst Scan Auto Scan Rate	Yes	Yes	Yes	ScanAsyst Auto Control
	ScanAsyst Auto Z Limit	Yes	Yes	Yes	ScanAsyst Auto Control

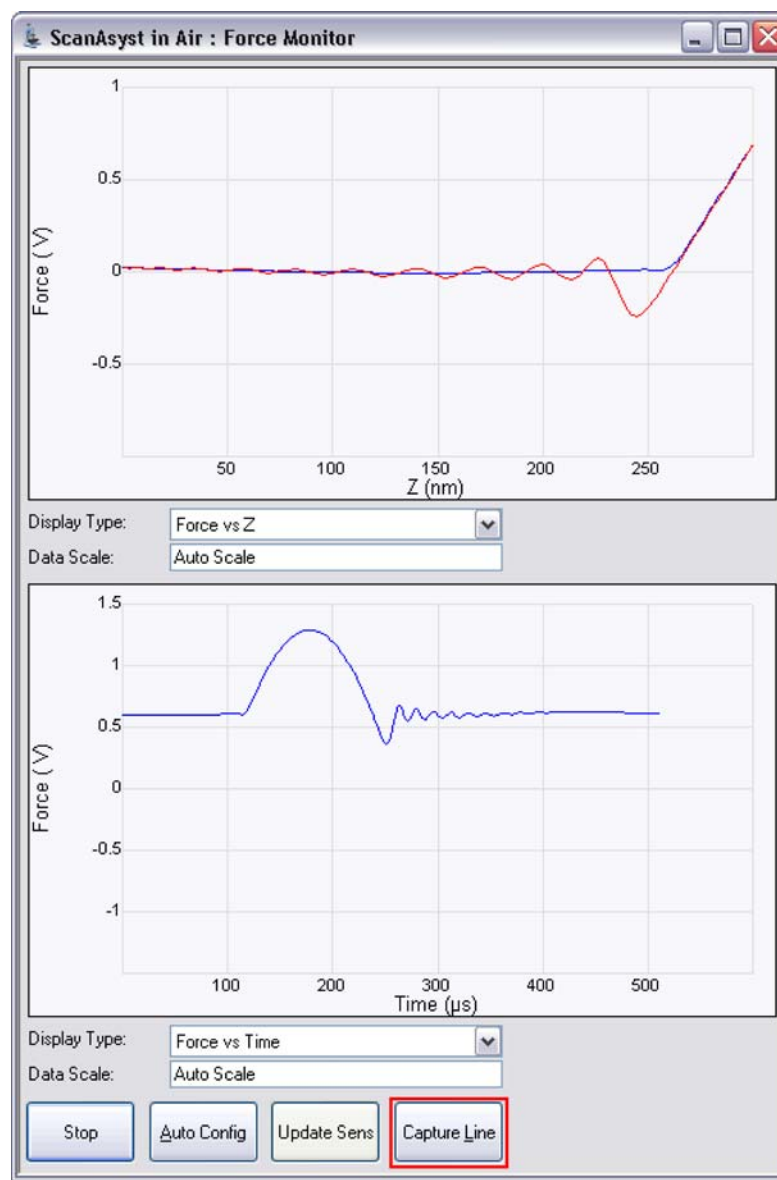
Panel	Parameter	Simple Mode	Expanded Mode	Show All	Other Dependencies
Peak Force QNM Control	Peak Force Amplitude	No	Yes	Yes	
	Lift Height	No	Yes	Yes	
	Top Fit Region	No	No	Yes	
	Unload Fit Region	No	No	Yes	
	Deformation Fit Region	No	No	Yes	
Cantilever Parameters	Spring Constant	Yes	Yes	Yes	
	Tip Radius	Yes	Yes	Yes	
	Poisson's Ratio	Yes	Yes	Yes	
Peak Force QNM Limits	Force Limit	No	Yes	Yes	
	Dissipation Limit	No	Yes	Yes	
	DMT Modulus Limit	Yes	Yes	Yes	
	LogDMT Modulus Limit	No	No	Yes	
	LogDMT Modulus Offset	No	No	Yes	
Limits	Z Limit	No	No	Yes	
	Z Range	Yes	Yes	Yes	
	Deflection Limit	No	Yes	Yes	

## 2.7 Capture Buttons

The capture buttons in the **Force Monitor** window allow you to collect data for use with the NanoScope Analysis off-line analysis software.

1. Start to collect a ScanAsyst/Peak Force Tapping image.
2. When you are in a region of interest, click the **CAPTURE LINE** button, shown in [Figure 2.7a](#), to capture a scan line.

**Figure 2.7a** CAPTURE LINE button



3. The **High Speed Data Capture** window, shown in [Figure 2.7b](#), will open and the **Status** will change when the data has been captured. **UPLOAD DATA** to the PC when the capture is complete. When **CAPTURE LINE** is used this way, the off-line NanoScope Analysis software will correctly associate the capture line of the high speed data capture with the line in the image.

**Figure 2.7b** High speed data capture is complete. However, the data is not immediately transferred to the PC.

**High Speed Data Capture**

**Channel Selection**

Rate: 6.25 MHz ChannelA Data Type: Off

Rate: 500 kHz ChannelB Data Type: Off

ChannelC Data Type: Deflection Error

ChannelD Data Type: Height

**Trigger Controls**

Arm Trigger ☐ Auto Re-Arm Event: EOL

Force Trigger ☒ Delay Upload Channel: Height

Level: 0.00 V

Slope: Positive

Delay: -512 ms

Duration: 1536 ms

Status: Failed

4. Click the **UPLOAD DATA** button to transfer the captured data to the computer. While the data transfer process takes place, the scan data will look corrupted because the DSP time is shared between PeakForce QNM properties computation and data transfer.



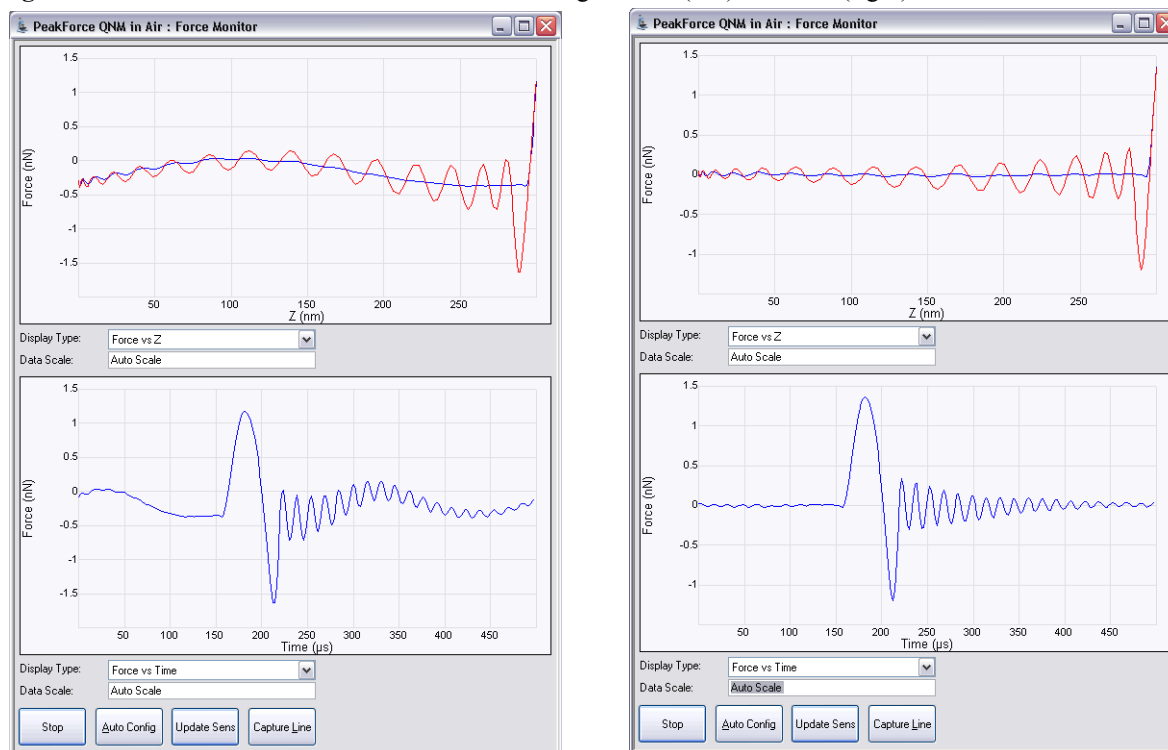
## 2.8 Optimizing a ScanAsyst Image

When the relative Z position between the probe and sample is modulated, parasitic cantilever motions occur. These motions include free-cantilever oscillation after snapping off the surface, deflection triggered by harmonics of the piezo motion or viscous forces. This parasitic deflection, defined as the deflection signal variation when the tip is NOT interacting with the sample, limits the low force range of ScanAsyst operation. Low force control is the most important factor to achieve high resolution imaging and property measurements.

During peak force tapping operation, the **Auto Config** operation is used to analyze the parasitic deflection signal including its data pattern by comparing the known source of parasitic excitation, namely the cantilever resonance at pulling off, modulation harmonics and other system actuation sources. The signature of the interaction, in the shape of heartbeat signal, is extracted from the parasitic deflections. The recovered heartbeat signal becomes the interaction force curve plotted in the time domain.

Figure 2.8a shows the heartbeat and force vs. Z curves of an image before and after **AUTO CONFIG** correction. The low frequency noise in the baseline has been removed.

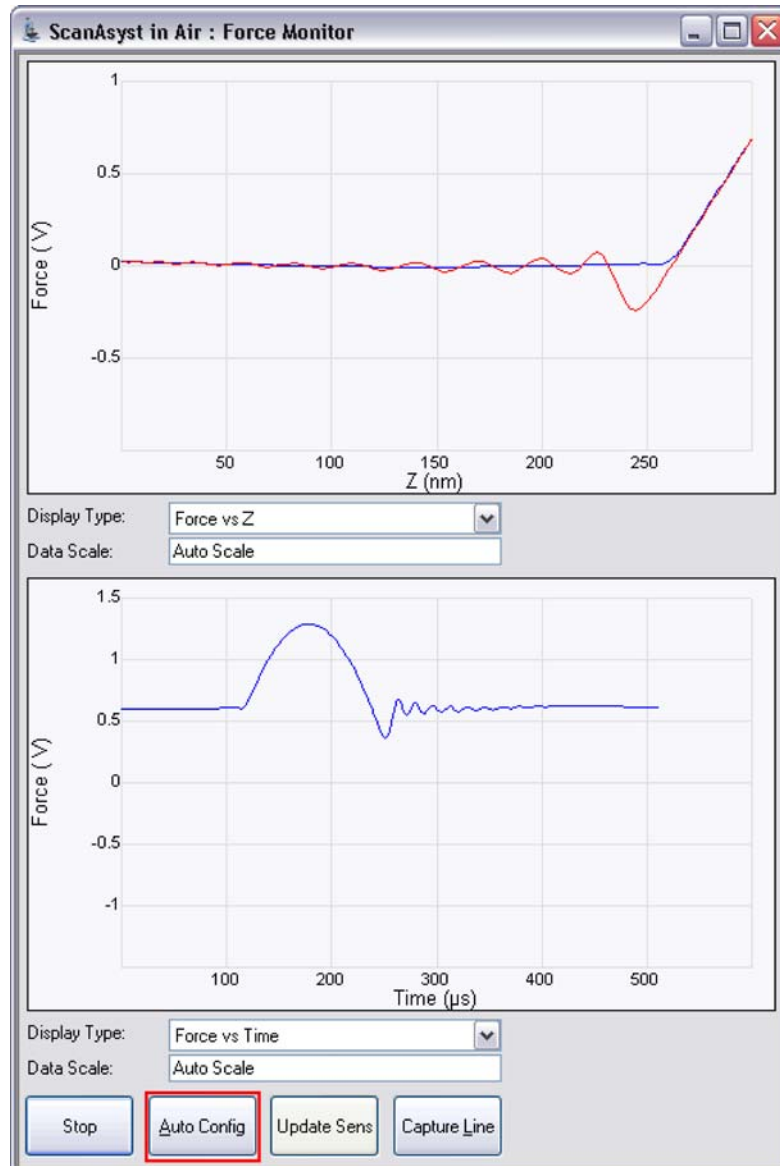
**Figure 2.8a** The heartbeat and force curves of an image before (left) and after (right) **AUTO CONFIG** correction.



If your force vs. time curves show parasitic background noise or the force vs. height curve load and unload curves are overlapping due to background noise, click the **AUTO CONFIG** button, shown in [Figure 2.8b](#), to invoke the real-time pattern analysis algorithm that removes parasitic deflection.

**Note:** Clicking **AUTO CONFIG** will automatically calculate the **Lift Height** and perform an **AUTO CONFIG** operation.

**Figure 2.8b** The AUTO CONFIG button



**Note:** When performing **AUTO CONFIG** operations with a small **Peak Force Setpoint** (less than ~20mV), the tip may drift out of contact with the surface and will be unable to return and track the surface. It is therefore recommended using a relatively large **Peak Force Setpoint** while performing **AUTO CONFIG** operations and reducing the **Peak Force Setpoint** later if necessary.

## 2.9 Advanced Atomic Force Operation

### 2.9.1 Displaying Parameters

You can adjust the number of parameters shown in the **Scan Parameter List** using several methods.

#### Simple Mode



1. The default **SIMPLE MODE**, intended for novice users and shown in [Figure 2.9a](#), displays the minimum number of parameters needed to make an image.

**Figure 2.9a** The **SIMPLE MODE** view of the **Scan Parameter List** for **PeakForce QNM in Air**

[-] Scan	
[-] Scan Size	500 nm
[-] Aspect Ratio	1.00
[-] X Offset	0.000 nm
[-] Y Offset	0.000 nm
[-] Scan Angle	0.00 °
[-] Scan Rate	0.977 Hz
[-] Samples/Line	512
[-] Feedback	
[-] Peak Force Setpoint	0.1000 V
[-] Feedback Gain	5.000
[-] ScanAsyst Auto Control	On
[-] Cantilever Parameters	
[-] Spring Constant	0.3000 N/m
[-] Tip Radius	10.0 nm
[-] Sample Poisson's Ratio	0.300
[-] PeakForce QNM Limits	
[-] DMT Modulus Limit	1024 Arb
[-] Limits	
[-] Z Range	7.42 µm
[-] Other	
[-] Units	Metric

## Expanded Mode



1. The **EXPANDED MODE** view, shown in [Figure 2.9b](#), increases the number of displayed parameters enabling expert users to fine tune an image.

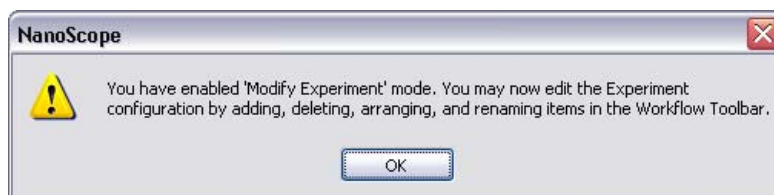
**Figure 2.9b** The **EXPANDED MODE** view of the **Scan Parameter List** for **ScanAsyst in Air**

[-] Scan	
Scan Size	500 nm
Aspect Ratio	1.00
X Offset	0.000 nm
Y Offset	0.000 nm
Scan Angle	0.00 °
Scan Rate	0.977 Hz
Tip Velocity	0.977 µm/s
Samples/Line	512
Lines	512
Slow Scan Axis	Enabled
XY Closed Loop	On
[-] Feedback	
Peak Force Setpoint	0.1000 V
Feedback Gain	5.000
LP Deflection BW	40.00 kHz
ScanAsyst Noise Threshold	0.500 nm
ScanAsyst Auto Control	On
[-] PeakForce QNM Control	
Peak Force Amplitude	150 nm
Lift Height	300 nm
[-] Cantilever Parameters	
Spring Constant	0.3000 N/m
Tip Radius	10.0 nm
Sample Poisson's Ratio	0.300
[-] PeakForce QNM Limits	
Force Limit	24.58 V
Dissipation Limit	1024 Arb
DMT Modulus Limit	1024 Arb
[-] Limits	
Z Range	7.42 µm
Deflection Limit	24.58 V
[-] Other	
LP Deflection	Enabled
Tip Bias Control	Ground
Sample Bias Control	Ground
Units	Metric
Minimum Engage Gain	10.0
Peak Force Engage Setpoint	0.1500 V
Bidirectional Scan	Disabled
Tip Serial Number	
Output 1 Data Type	Off
Output 2 Data Type	Off

## Show All

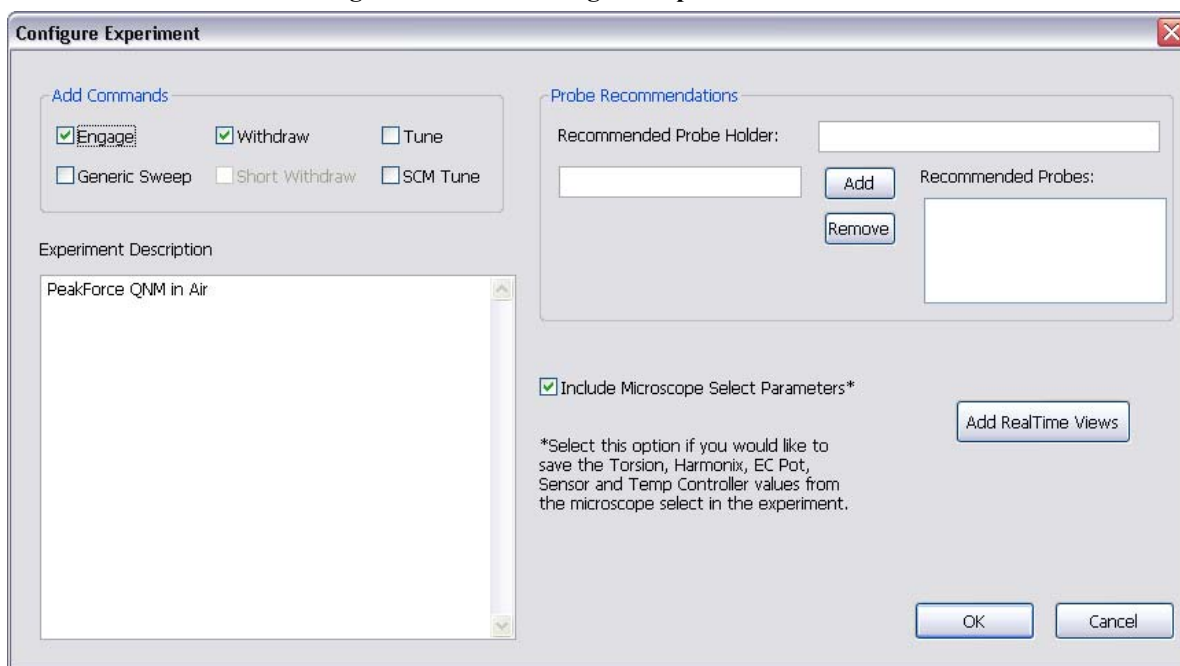
1. From the Menu bar, click **EXPERIMENT > CONFIGURE EXPERIMENT**. This opens an information window, shown in [Figure 2.9c](#).

**Figure 2.9c** The Configure Experiment information window



2. Click **OK** to open the **Configure Experiment** window, shown in [Figure 2.9d](#).

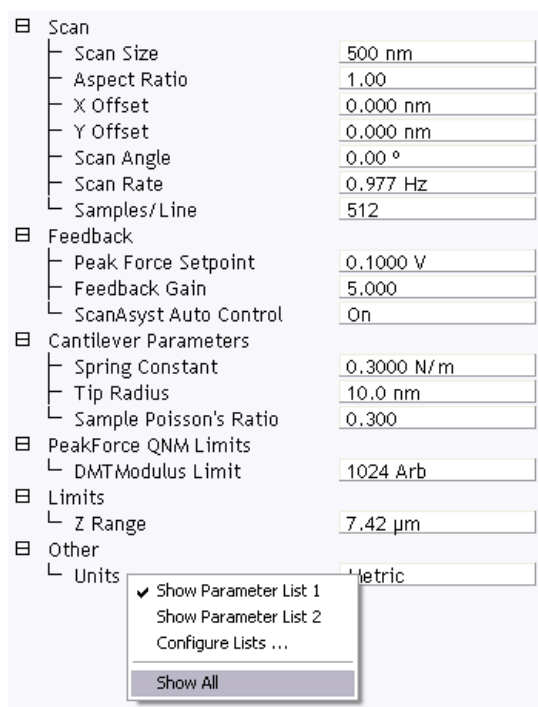
**Figure 2.9d** The Configure Experiment Window



3. Check a box in the **Add Commands** panel to add that command to the **Workflow Toolbar**.
4. Click **OK** to accept your choices and close the **Configure Experiment** window.

- Right-click in the **Scan Parameter List** and select **SHOW ALL**, shown in [Figure 2.9e](#).

**Figure 2.9e** Select **SHOW ALL** items



This makes all **Scan Parameters** visible along with two check boxes, the left, green, check box for the **SIMPLE MODE** and the right, red, check box for the **EXPANDED MODE**. See [Figure 2.9f](#).

**Figure 2.9f** Enable Parameters

With "✓" Parameter will display

Without "✓" Parameter will not display

Group	Parameter	Value
Scan	Scan Size	500 nm
	Aspect Ratio	1.00
	X Offset	0.000 nm
	Y Offset	0.000 nm
	Scan Angle	0.00 °
	Scan Rate	0.977 Hz
	Tip Velocity	0.977 µm/s
	Samples/Line	512
	Lines	512
	Slow Scan Axis	Enabled
Feedback	XY Closed Loop	On
	SPM Feedback	Peak Force
	Lateral 16x Gain	Disabled
	Peak Force Setpoint	0.1000 V
	Feedback Gain	5.000
	Proportional Gain	0
	Analog3	0 V
	Analog4	0 V
	LP Deflection BW	40.00 kHz
	ScanAsyst Setup	Allow
PeakForce QNM Control	ScanAsyst Noise Threshold	0.500 nm
	ScanAsyst AutoConfig Fram	0
	ScanAsyst Auto Control	On
	Peak Force Amplitude	150 nm
	Lift Height	300 nm
Cantilever Parameters	Top Fit Region	10 %
	Unload Fit Region	70 %
	Deformation Fit Region	85 %
	Spring Constant	0.3000 N/m
PeakForce QNM Limits	Tip Radius	10.0 nm
	Sample Poisson's Ratio	0.300
	Force Limit	24.58 V
Limits	Dissipation Limit	1024 Arb
	DMTModulus Limit	1024 Arb
	LogDMTModulus Limit	32.00 log(Arb)
	LogDMTModulus Offset	0 log(Arb)
Other	Z Limit	8.957 µm
	Z Range	7.42 µm
	Deflection Limit	24.58 V

☒ Show Parameter List 1  
☒ Show Parameter List 2  
☐ Configure Lists ...  
☒ Show All

The checked ✓ parameters display in normal Real-time mode while those parameters without a ✓ will not display in normal Real-time mode.

Check the parameters that you want displayed and right-click in the **Scan Parameter List** and select **SHOW ALL** items to hide the unchecked parameters. The panel will once again appear in normal Real-time mode.





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## Chapter 3    PeakForce QNM Samples

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Five samples are supplied with the PeakForce QNM kit.

**Note:** All samples are homogeneous and glued to Bruker sample pucks.

**Note:** Probe or sample contamination may compromise the accuracy of the quantitative measurements.

### 3.1 PDMS-Soft-1

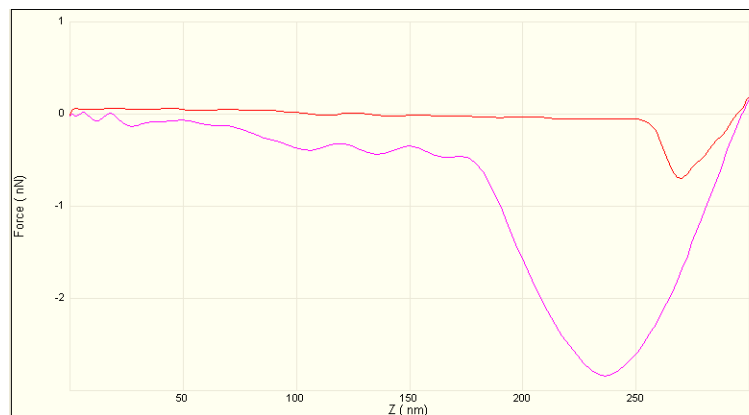
Nominal modulus: 2.5 MPa.

This 150  $\mu\text{m}$  thick PDMS gel sample is formulated for long shelf life and stability.

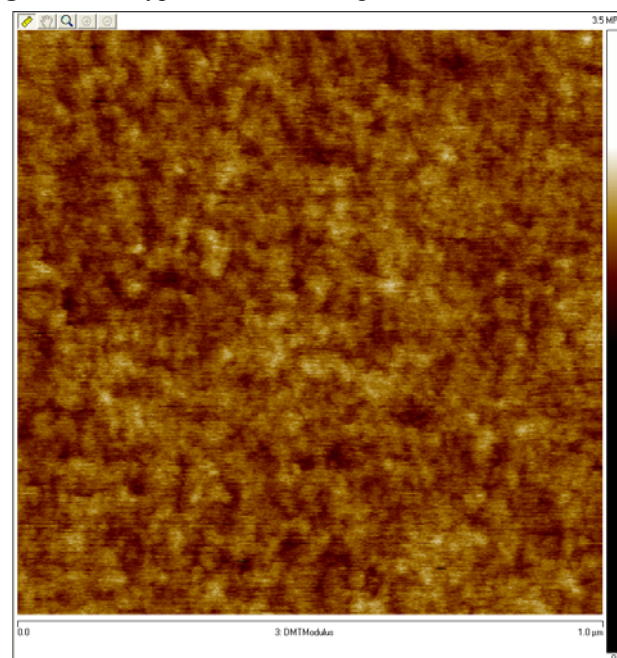
Suggested probe: ScanAsyst-Air.

A typical force curve of this sample is shown in [Figure 3.1a](#) and an image of the modulus is shown in [Figure 3.1b](#). The standard deviation of the modulus in this image is 0.7 MPa.

**Figure 3.1a** Typical force curve of a PDMS-Soft-1 sample



**Figure 3.1b** Typical modulus image of a PDMS-Soft-1 sample



## 3.2 PDMS-Soft-2

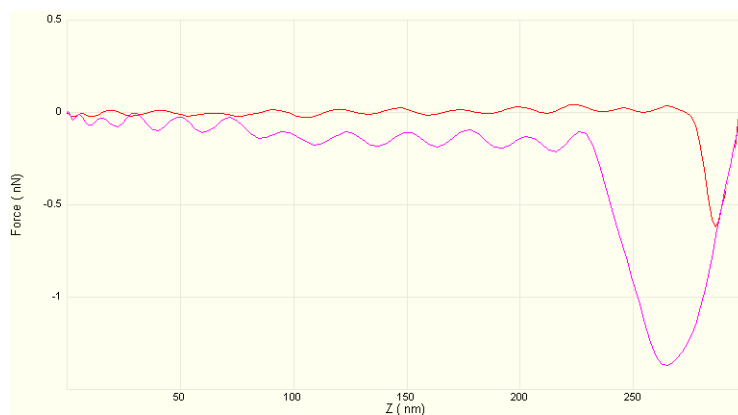
Nominal modulus: 3.5 MPa.

This 150  $\mu\text{m}$  thick PDMS gel sample is formulated for long shelf life and stability.

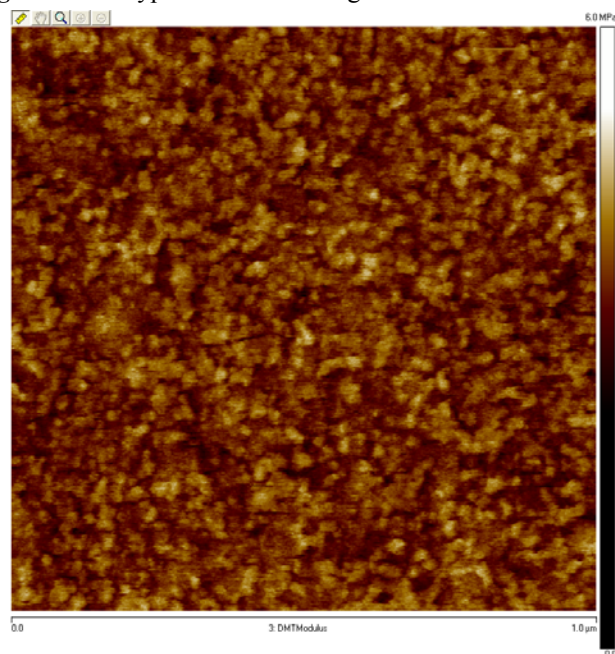
Suggested probe: ScanAsyst-Air.

A typical force curve of this sample is shown in [Figure 3.2a](#) and an image of the modulus is shown in [Figure 3.2b](#). The standard deviation of the modulus in this image is 0.5 MPa.

**Figure 3.2a** Typical force curve of a PDMS-Soft-2 sample



**Figure 3.2b** Typical modulus image of a PDMS-Soft-2 sample



### 3.3 Polystyrene

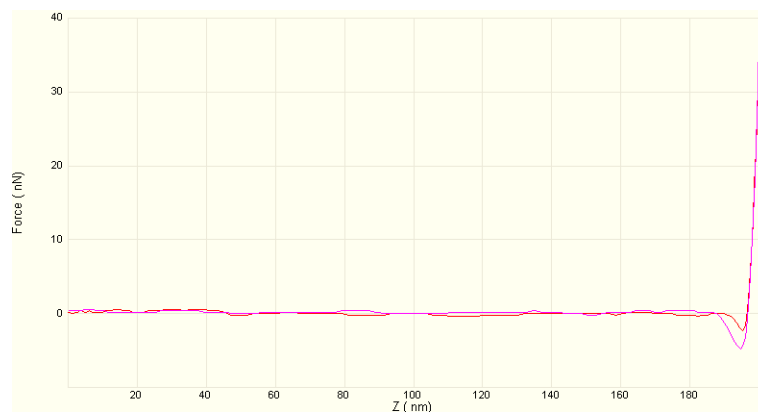
Nominal modulus 2.7 GPa.

This polystyrene sample is spin-cast on a silicon wafer.

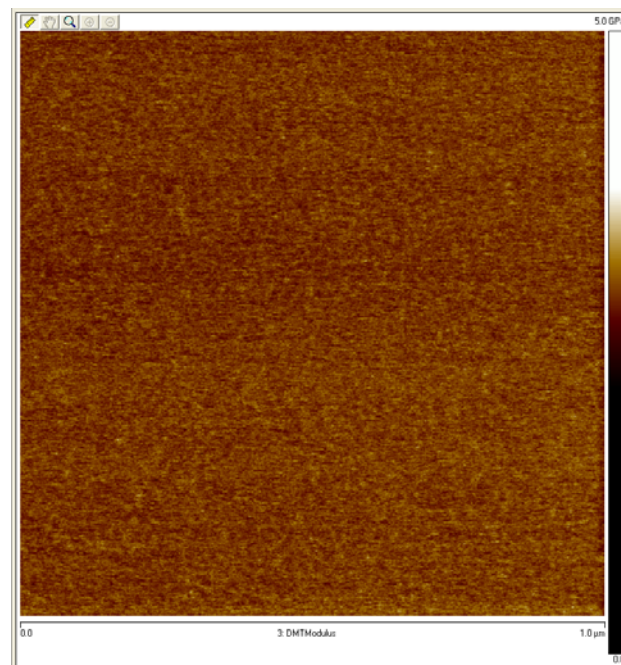
Suggested probes: RTESPA/Tap300 or Tap525.

A typical force curve of this sample is shown in [Figure 3.3a](#) and an image of the modulus is shown in [Figure 3.3b](#). The standard deviation of the modulus in this image is 0.2 MPa.

**Figure 3.3a** Typical force curve of a Polystyrene sample



**Figure 3.3b** Typical modulus image of a Polystyrene sample



### 3.4 HOPG

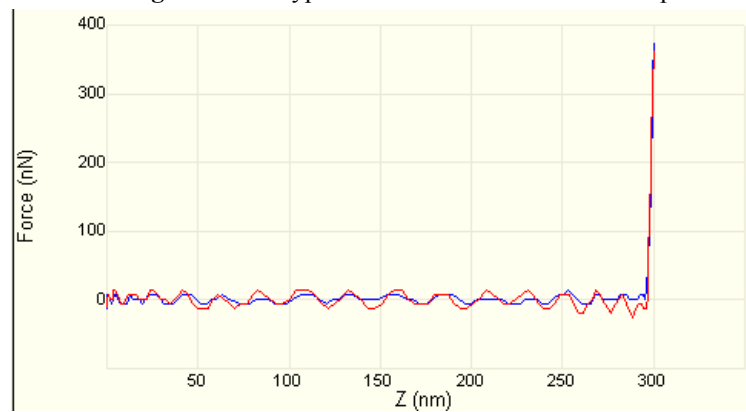
Highly Oriented Pyrolytic Graphite.

Nominal modulus 18 GPa.

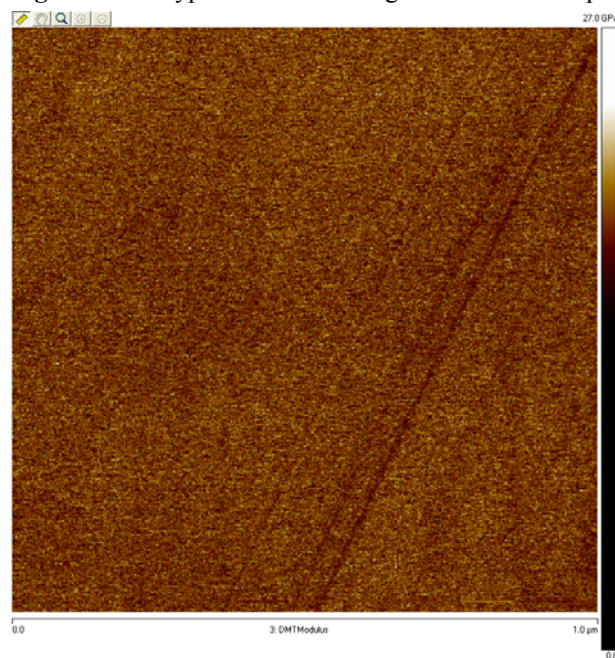
Suggested probes: TESPA, diamond

A typical force curve of this sample is shown in [Figure 3.4a](#) and an image of the modulus is shown in [Figure 3.4b](#). The standard deviation of the modulus in this image is 2 MPa.

**Figure 3.4a** Typical force curve of a HOPG sample



**Figure 3.4b** Typical modulus image of a HOPG sample



### **3.5 Fused Silica**

Corning 7980 fused silica.

Nominal modulus 72.9 GPa.

Suggested probe; Diamond.

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# Chapter 4 Calibration

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## 4.1 Introduction to Calibrating PeakForce QNM

To quantify the forces as well as other mechanical properties of your sample, it is important to understand the PeakForce QNM calibration procedure.

**Note:** For best results, the calibration process will need to be performed for each probe.

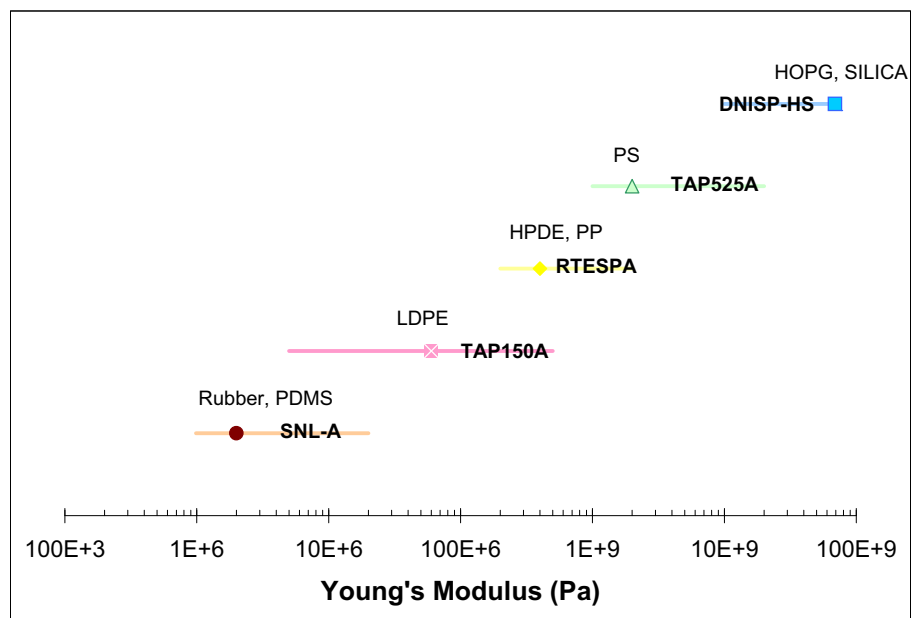
## 4.2 Absolute vs. Relative Calibration Methods

There are two methods of obtaining calibrated, quantitative results from PeakForce QNM. The first method (the relative method) avoids accumulated errors that can cause errors in modulus measurements, but has the downside in that it requires a reference sample that can be measured by the same probe as the unknown sample. The second method (the absolute method) does not require a reference sample, but requires accurate measurement of the tip end radius (typically by scanning an artifact sample like TipCheck) and spring constant (typically with thermal tune for soft cantilevers). Both methods require measurement of the deflection sensitivity on a hard sample.

For both methods, it is important to choose a probe that can cause enough deformation in the sample and still retain high force sensitivity. [Figure 4.2a](#) shows the recommended probes and the modulus range over which they work best.



**Figure 4.2a** Modulus ranges covered by various probes. The modulus of the reference sample for each range is indicated as well.



**Table 4.2a** Legend

Symbol	Chemical Name
PDMS	Polydimethylsiloxane
LPDE	Low-density polyethylene
HPDE	High-density polyethylene
PP	Polypropylene
PS	Polystyrene
HOPG	Highly Oriented Pyrolytic Graphite

### 4.2.1 The Relative Method

The relative method of calibration uses a sample of known modulus to obtain the ratio of spring constant to the square root of tip end radius. It is still important to accurately calibrate the deflection sensitivity in order to obtain modulus results. An outline of the procedure follows:

1. Calibrate the **Deflection Sensitivity** on a clean, hard sample (Sapphire or Silicon, which can be used for samples with modulus less than 10 GPa). See **Calibrate the Deflection Sensitivity**: [Section 4.3](#) for a procedure to measure this.
2. If quantitative **Adhesion** or **Dissipation** data is required, use the NanoScope **Thermal Tune** function to obtain the spring constant, otherwise enter the nominal value from the manufacturer. See **Calibrate the Spring Constant Using Thermal Tuning**: [Section 4.4](#) for a procedure to measure this.
3. Image the reference sample using **PeakForce QNM** and adjust the **Tip Radius** parameter to make the measured **Modulus** equal the known value of the reference sample.
4. Image the unknown sample adjusting the **Peak Force Setpoint** to match the deformation depth used during imaging of the reference sample.

### 4.2.2 The Absolute Method

The absolute procedure is very similar to the relative procedure except for two important differences:

1. The spring constant calibration ([Step 2](#)) is not optional.
2. Instead of using the reference sample, the tip end radius is measured by scanning a tip calibration artifact (such as TipCheck Bruker part #RS) and analyzing the resulting image. See **Measure the Tip Radius**: [Section 4.5](#) for a procedure to measure this.

**Note:** The absolute procedure has the benefit that there is no concern over the accuracy of the modulus of the reference sample or whether it ages over time or becomes contaminated.

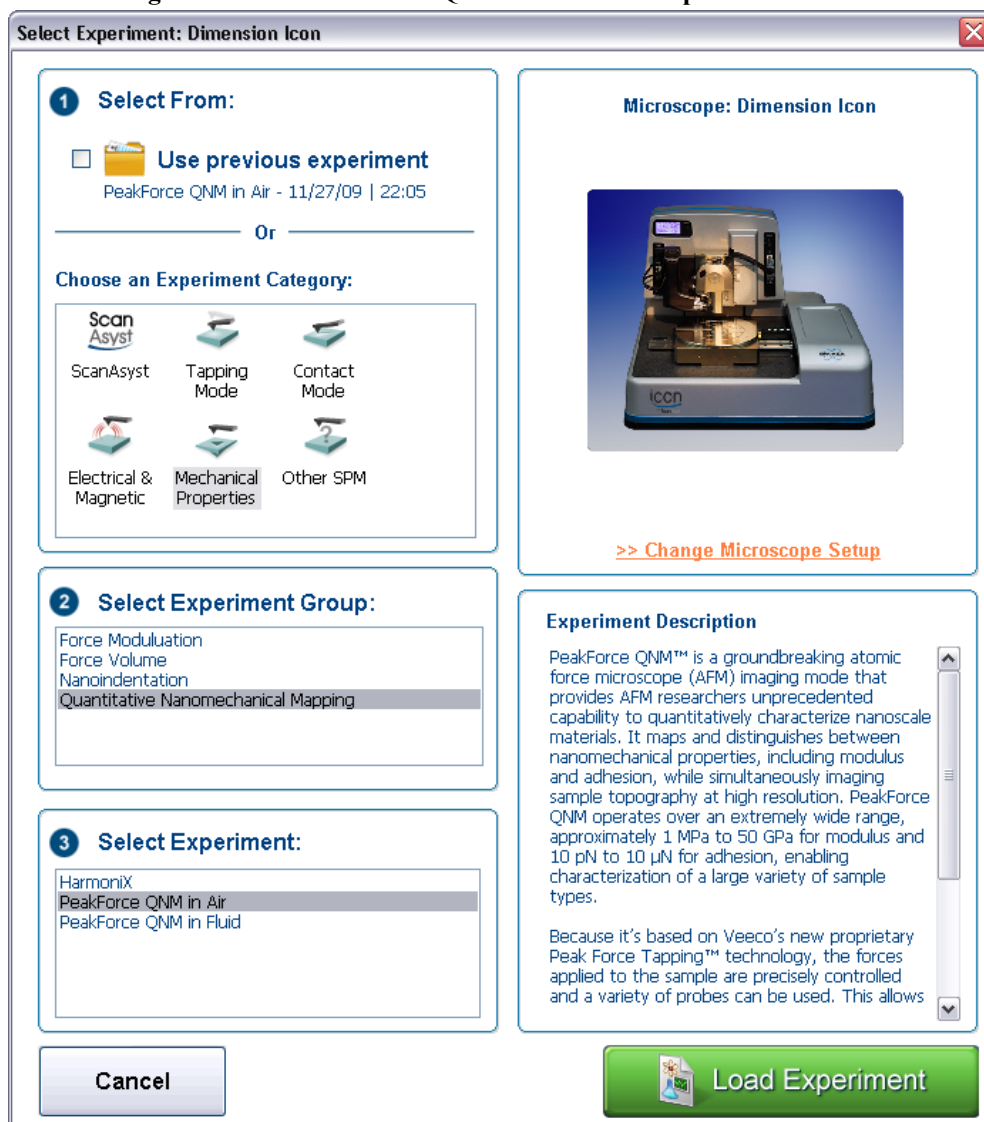
## 4.3 Calibrate the Deflection Sensitivity

Because Peak Force QNM mode ramps the Z piezo and acquires force curves, measuring deflection sensitivity requires fewer steps than the normal ramp procedure.



1. Click the **SELECT EXPERIMENT** icon. This opens the **Select Experiment** window, shown in [Figure 4.3a](#).

**Figure 4.3a** The PeakForce QNM in Air **Select Experiment** window



2. Select **MECHANICAL PROPERTIES** in the **Experiment Category** panel.
3. Select **QUANTITATIVE NANOMECHANICAL MAPPING** in the **Select Experiment Group** panel.

4. Select **PEAKFORCE QNM IN AIR** in the **Select Experiment** panel and click **LOAD EXPERIMENT**.

5. Set the **Scan Size** to **0 nm**.



6. **ENGAGE** the probe onto a clean sapphire (required for cantilevers with  $k > 200$  N/m) or silicon surface using the **PeakForce QNM in Air** mode.



7. Activate **RAMP** mode by clicking the **RAMP** icon in the **Workflow Toolbar**. This causes the system to stop scanning, and the probe to position above the center of the previous image.

8. Enter the following parameter settings in the designated panels of the **Ramp Parameter List**:

- a. In the **Ramp** panel select:

Parameter	Setting
Ramp output	Z
Ramp size	100nm - 1.00μm
Scan Rate	1.00Hz
Number of samples	512

- b. In the **Mode** panel select:

Parameter	Setting
Trigger mode	Relative
Trig threshold	0.2V

- c. In the **Channel 1** panel select:

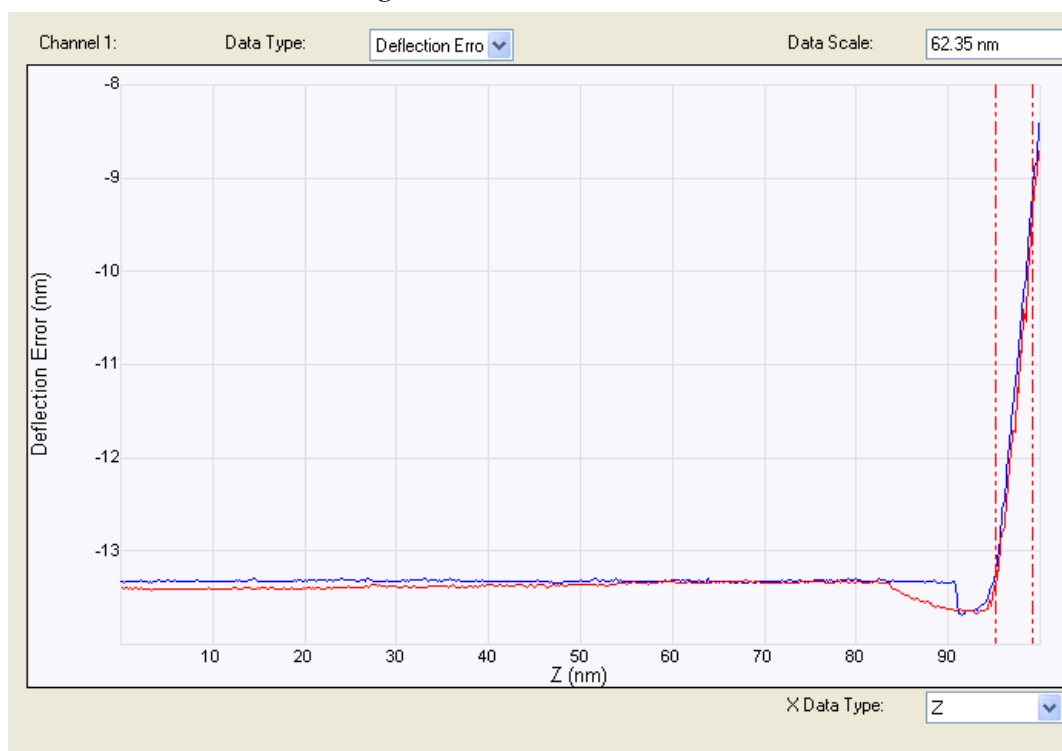
Parameter	Setting
Data Type	Deflection Error
X Data Type	Z
Display Mode	Deflection Error vs. Z



9. Click the **RAMP SINGLE** icon on the NanoScope toolbar or select **Ramp > Ramp Single**, from the menu bar.

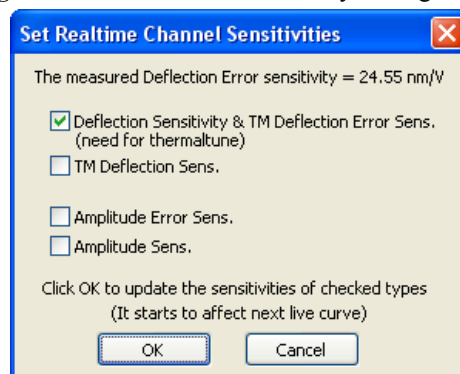
10. Move two cursors onto the Deflection vs. Z plot (see [Figure 4.3b](#)).
11. Arrange the cursors so that they surround the contact (steepest) portion of the graph (see [Figure 4.3b](#)).

**Figure 4.3b** Force Curve Cursors



12. Click the **UPDATE SENSITIVITY** icon in the NanoScope toolbar or select **Ramp > Update Sensitivity**. The software will automatically calculate the deflection sensitivity and open the Set Realtime Channel Sensitivities window (see [Figure 4.3c](#)).

**Figure 4.3c** Deflection Sensitivity Dialogue Box



13. Click **OK** to accept this deflection sensitivity in the dialogue box that displays, and it will automatically be entered into the **Deflection Sensitivity** parameter.

## 4.4 Calibrate the Spring Constant Using Thermal Tuning

**Note:** Bruker recommends **Thermal Tune** included in NanoScope software for probes with spring constants less than or equal to 1 N/m. Other methods (Sader<sup>1</sup>, added mass, vibrometer, pre-calibrated probes) are recommended for probes with higher spring constants. These techniques are reviewed in detail in Bruker Application Note 94: *Practical Advice on the Determination of Cantilever Spring Constants*.



1. Ensure that the probe is withdrawn adequately from the sample before activating **THERMAL TUNE**. The probe should not interact with the sample during its self excitation under ambient conditions.
2. Click **CALIBRATE > THERMAL TUNE** or the **THERMAL TUNE** icon in the NanoScope tool bar (shown).
3. Select a frequency range that includes the resonant frequency of the cantilever. See [Figure 4.4a](#). Stiff cantilevers may require the 5 - 2000 kHz range.

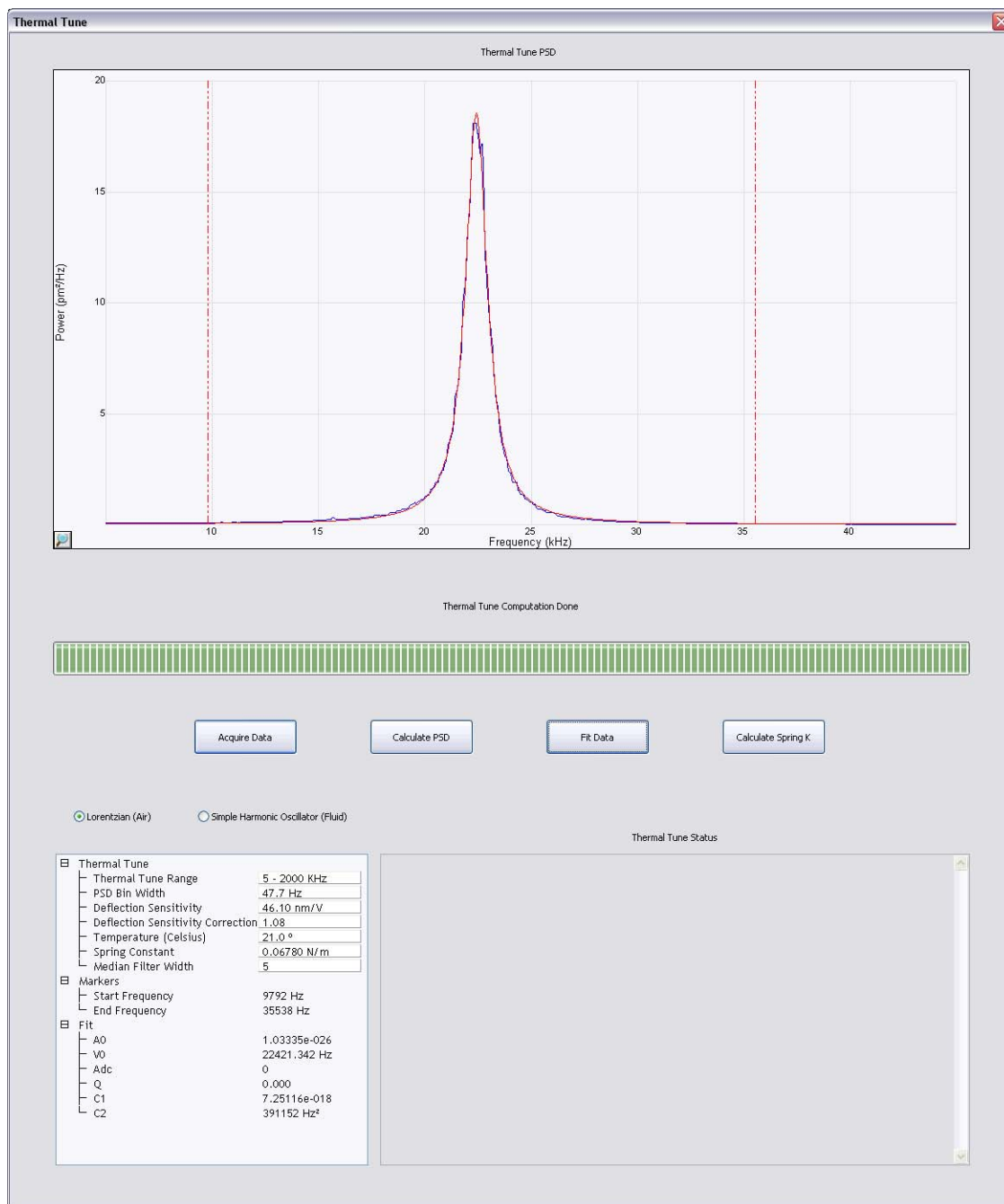
**Figure 4.4a** Select Thermal Tune Frequency Range

Thermal Tune	
Thermal Tune Range	1 - 100 KHz
PSD Bin Width	1 - 100 KHz
Deflection Sensitivity	5 - 2000 KHz
Deflection Sensitivity Corre	1.08
Temperature (Celsius)	21.0 °
Spring Constant	0.06780 N/m
Median Filter Width	3

1. See <http://www.ampc.ms.unimelb.edu.au/afm/calibration.html>

- Click **ACQUIRE DATA** in the **Thermal Tune** panel, shown in [Figure 4.4b](#).

**Figure 4.4b** The **Thermal Tune** panel



- The microscope will acquire data for about 30 seconds.
- Zoom in on the region around the peak.

7. Click either the **LORENTZIAN (AIR)** or **SIMPLE HARMONIC OSCILLATOR (FLUID)** button to select a Lorentzian or a simple harmonic oscillator model, respectively, of the PSD to be least squares fit to the data.

**Note:** The equations used to fit the filtered data are:

Lorentzian, for use in air

$$A(\nu) = A_0 + \frac{C_1}{(\nu - \nu_0)^2 + C_2}$$

**Note:** where:  $A(\nu)$  is the amplitude as a function of frequency,  $\nu$   
 $A_0$  is the baseline amplitude  
 $\nu_0$  is the center frequency of the resonant peak  
 $C_1$  is a Lorentzian fit parameter  
 $C_2$  is a Lorentzian fit parameter

Simple Harmonic Oscillator, for use in fluid

$$A(\nu) = A_0 + A_{DC} \cdot \frac{\nu_0^2}{\sqrt{(\nu_0^2 - \nu^2)^2 + \frac{\nu_0^2 \nu^2}{Q^2}}}$$

where:  $A(\nu)$  is the amplitude as a function of frequency,  $\nu$   
 $A_0$  is the baseline amplitude  
 $A_{DC}$  is the amplitude at DC (zero frequency)  
 $\nu_0$  is the center frequency of the resonant peak  
 $Q$  is the quality factor

8. Adjust the **Median Filter Width**, shown in [Figure 4.4c](#), to remove individual (narrow) spikes. This replaces a data point with the median of the surrounding  $n$  ( $n=3, 5, 7$ ) data points.

**Figure 4.4c Median Filter Width**

The screenshot shows the 'Thermal Tune' settings window. Under the 'Thermal Tune' section, the 'Median Filter Width' is set to 3. Other settings include Thermal Tune Range (1 - 100 KHz), PSD Bin Width (7.63 Hz), Deflection Sensitivity (60.00 nm/V), Deflection Sensitivity Corre (1.08), Temperature (Celsius) (21.0 °), Spring Constant (0.06780 N/m), and Markers (Disable). The Start Frequency is 3 and the End Frequency is 7.

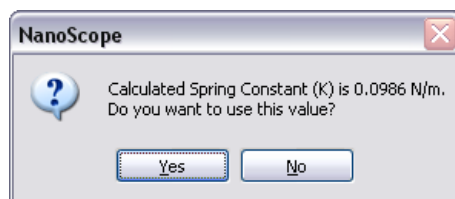
Thermal Tune	
Thermal Tune Range	1 - 100 KHz
PSD Bin Width	7.63 Hz
Deflection Sensitivity	60.00 nm/V
Deflection Sensitivity Corre	1.08
Temperature (Celsius)	21.0 °
Spring Constant	0.06780 N/m
Median Filter Width	3
Markers	
Start Frequency	3
End Frequency	7

9. Adjust the **PSD Bin Width** to reduce the noise by averaging.



10. Drag markers in from the left and/or right plot edges to bracket the bandwidth over which the fit is to be performed. See [Figure 4.4b](#).
11. Click **FIT DATA**. The curve fit, in red, is displayed along with the acquired data. If necessary, adjust the marker positions and fit the data again to obtain the best fit at the thermal peak.
12. Enter the cantilever **Temperature**.
13. Click **CALCULATE SPRING K**.
14. You will be asked whether you want to accept the calculated value of the spring constant,  $k$  (see [Figure 4.4d](#)). Clicking **OK** copies the calculated spring constant to the **Spring Constant** window in the **Cantilever Parameters** window.

**Figure 4.4d** Spring Constant Calculation Result

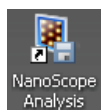


## 4.5 Measure the Tip Radius

Tip radius may be measured using a tip characterizer sample and the **Tip Qualification** function in NanoScope Analysis software (NanoScope software does not include the Estimated End Radius function).



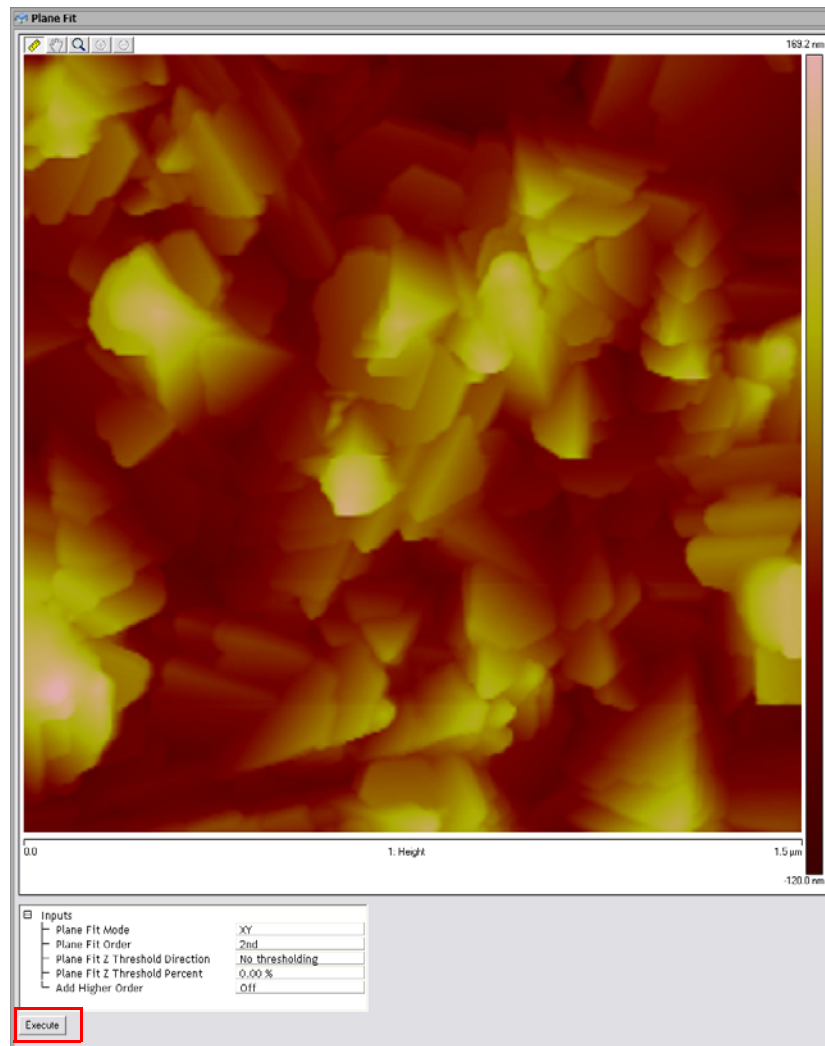
1. **Scan (PEAK FORCE QNM IN AIR mode)** the characterizer sample. Set the **Scan Size** to approximately 1.5  $\mu\text{m}$ . Characterizer image size is important because, along with **Tip Image Size** and feature density, it determines how many peaks are used for the tip estimation.
2. Set the **Samples/Line** and **Lines** to **512**.
3. Set the **Aspect Ratio** to **2.0**
4. Set the **Scan Rate** to **0.5 Hz** or less.
5. Because this is an intentionally rough sample that can damage the probe tip, set **ScanAsyst Noise Threshold** to **1.0 nm**.
6. **CAPTURE** the characterizer image.
7. Open the **Height** channel of the saved image in the **NANOSCOPE ANALYSIS** package.





8. Flatten the image by clicking the **PLANE FIT** icon.
9. Select **XY** as the **Plane Fit Mode**.
10. Select **1st** AS the **Plane Fit Order**.
11. Click **EXECUTE** to plane fit the image. See [Figure 4.5a](#).

**Figure 4.5a** Plane Fit of the Characterizer Sample

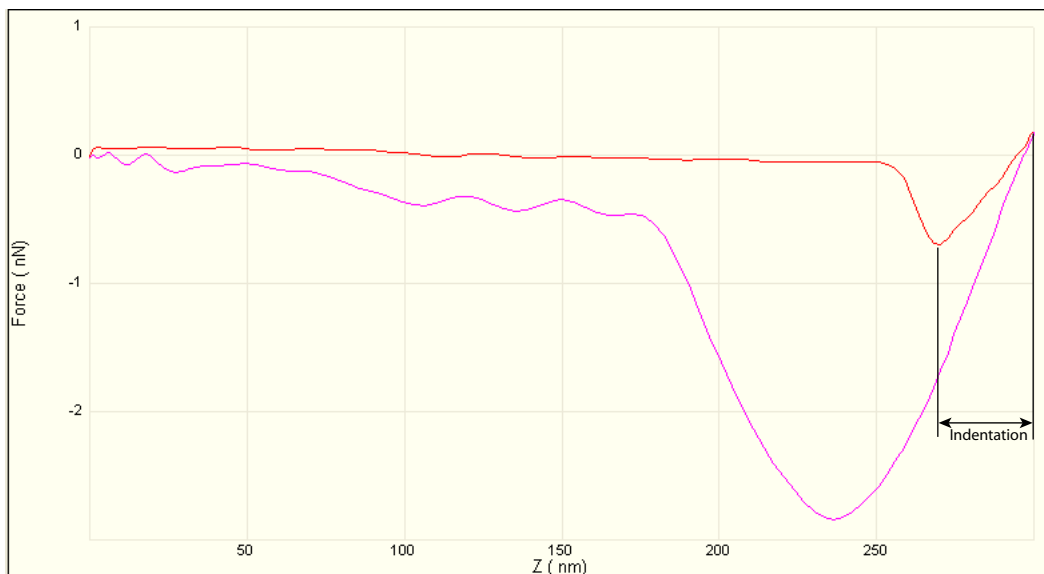




12. Click the **TIP QUALIFICATION** icon in the **NanoScope Analysis** toolbar to open the **Tip Qualification** window.
13. Enter the measured average **Deformation** (see **Deformation**: [Section 2.5.6](#)) into the **Height 1 from Apex** field.

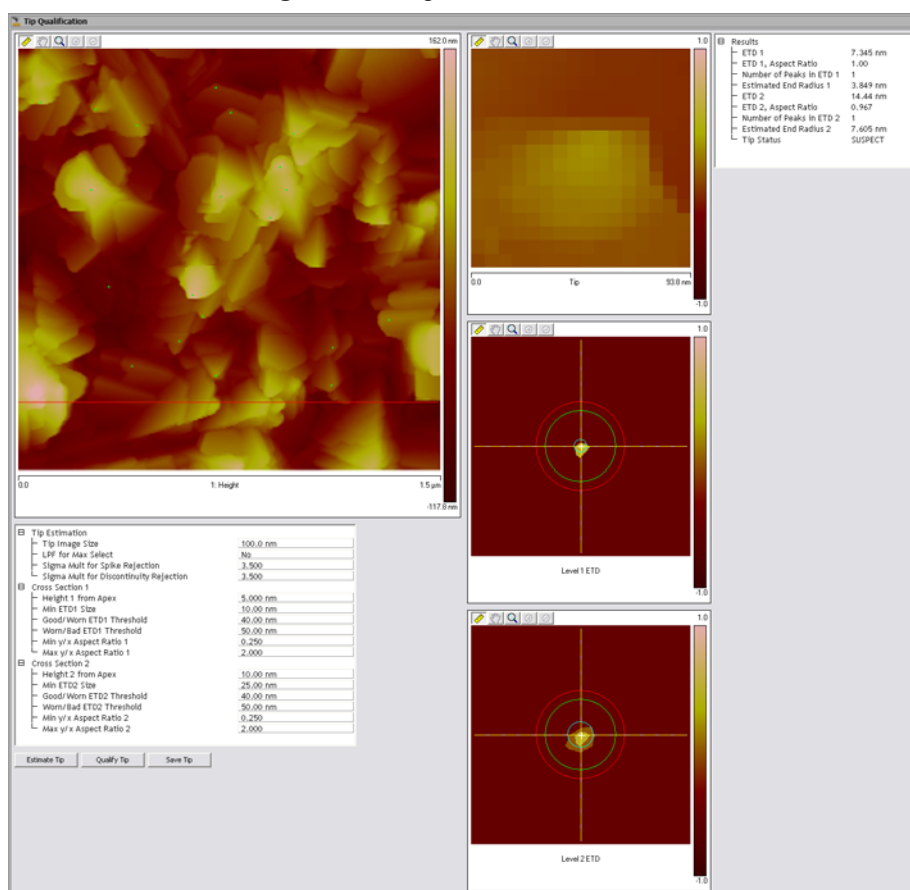
**Note:** The **Height from Apex** parameter should equal the average penetration depth or the indentation in the force curve. The indentation can be measured as the separation from the minimum force to the peak force in the loading curve. For most samples, the indentation is very close to the sample deformation. Therefore, the average deformation can be used as **Height from Apex** parameter to estimate tip radius. But for very soft samples (<20MPa), the adhesion is very large and the difference between indentation and deformation is large so that the indentation must be measured from the force curves in the **Force Monitor** window shown in [Figure 4.5b](#).

**Figure 4.5b** Typical force curve of a PDMS-Soft-1 sample. Nominal modulus: 2.5 MPa.



14. Click **ESTIMATE TIP**.
15. Click **QUALIFY TIP**. [Figure 4.5c](#) displays the **Estimated Tip End Radius**.

Figure 4.5c Tip Qualification Results



## 4.6 Calibrate Peak Force QNM

Three parameters are needed to fully calibrate PeakForce QNM:

1. **Deflection Sensitivity.** See **Calibrate the Deflection Sensitivity:** [Section 4.3](#) for a procedure to measure this.
2. **Spring Constant.** See **Calibrate the Spring Constant Using Thermal Tuning:** [Section 4.4](#) for a procedure to measure this.
3. **Tip Radius.** See **Measure the Tip Radius:** [Section 4.5](#) for a procedure to measure this.

A fourth parameter, the **Sample's Poisson's Ratio**, is needed to convert the measured reduced modulus,  $E^*$ , to the sample modulus,  $E_s$ . The reduced modulus is related to the sample modulus by the following equation:

$$E^* = \left[ \frac{1 - \nu_t^2}{E_t} + \frac{1 - \nu_s^2}{E_s} \right]^{-1}$$

where  $\nu_t$  and  $E_t$  are the Poisson's ratio and Young's modulus of the tip and  $\nu_s$  and  $E_s$  are the Poisson's ratio and Young's modulus of the sample. We assume that the tip modulus,  $E_t$ , is much larger than the sample modulus,  $E_s$ , and can be approximated as infinite and calculate the sample modulus using the sample Poisson's Ratio

Poisson's ratio generally ranges between about 0.2 and 0.5 (perfectly incompressible) giving a difference between the reduced modulus and the sample modulus between 4% and 25%. Because the sample's Poisson's ratio is not generally known, many publications report only the reduced modulus. Entering zero for this parameter will cause the system to return the reduced modulus. Recommended values for the sample's Poisson ratio,  $\nu_s$ , are shown in [Table 4.6a](#).

**Table 4.6a** Recommended values of the sample Poisson's ratio,  $\nu_s$ , as a function of the sample stiffness,  $E_s$ .

$E_s$	$\nu_s$
$E_s < 100$ MPa	0.5
$0.1 < E_s < 1$ GPa	0.4
$1 \text{ GPa} < E_s < 10$ GPa	0.3

### 4.6.1 Cantilever Parameters

After you have measures the cantilever **Spring Constant** and the **Tip Radius**, enter them into the **Cantilever Parameters** panel in the Scan Parameters window of the NanoScope software window, shown in [Figure 4.6a](#). If available, enter the **Sample Poisson's Ratio**.

**Figure 4.6a** The Cantilever Parameters panel

Figure 4.6a shows the 'Cantilever Parameters' panel in the NanoScope software. The panel contains three input fields: 'Spring Constant' with a value of 0.3000 N/m, 'Tip Radius' with a value of 10.0 nm, and 'Sample Poisson's Ratio' with a value of 0.300.

### 4.6.2 Feedback Parameters

#### Peak Force Setpoint

A **Peak Force Setpoint** that is too high can either damage the sample or wear the tip. It is generally desirable to reduce the **Peak Force Setpoint** to as small a value as is possible. However, in order to achieve accurate Elastic modulus measurement, sufficient sample deformation is needed. If the deformation is less than 2nm, increase the **Peak Force Setpoint** to achieve sufficient sample deformation.

For the relative method, you should adjust the **Peak Force Setpoint** to keep the **Deformation** the same for both the reference and measurement samples.

### **Feedback Gain**

Reduce the **Feedback Gain** to lower the noise in the property channels.

If **ScanAsyst Auto Gain** is On, set the **ScanAsyst Noise Threshold** to 0.5nm or less.



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# Chapter 5    Offline Analysis

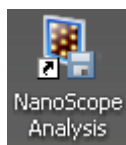
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## 5.1 Introduction

As discussed in [Chapter 2](#), real-time PeakForce QNM saves images of processed data like modulus and adhesion. There are times when one would like to compare these processed data images with the associated force curves. For this reason, a PeakForce QNM mode off-line analysis function is included in the NanoScope Analysis package. The main intention of this function is to allow you to view and analyze force curves from areas where material properties are most likely to change. You are given options to export raw force curves that can then be analyzed in the NanoScope Analysis or third party analysis programs.

As discussed in [Section 2.7](#), you should collect an image and then, at a region of interest during the capture, click the **CAPTURE LINE** button. This ensures capture of the raw high speed data capture (HSDC) in the DSP buffers. Remember that to transfer the data to the computer and into a file, you must click the **UPLOAD DATA** button in the **High Speed Data Capture** interface.

## 5.2 Procedure



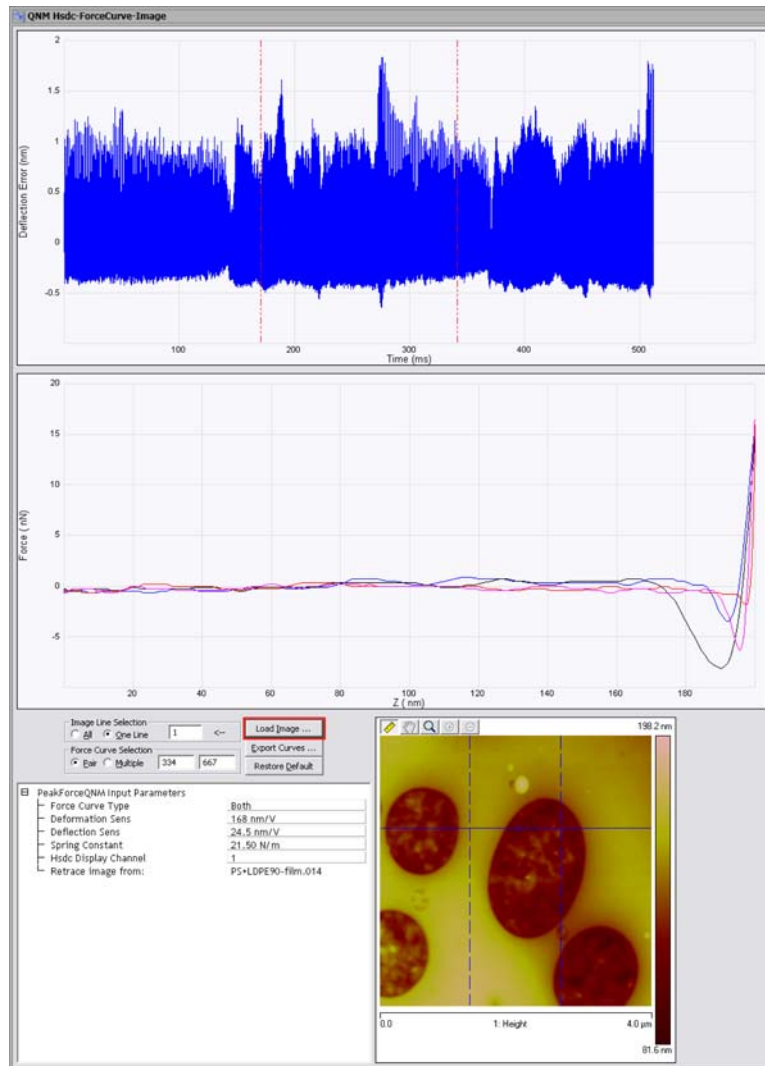
1. Start the NanoScope Analysis package by double clicking the offline icon on the Windows desktop.
2. Open the PeakForce QNM HSDC file.





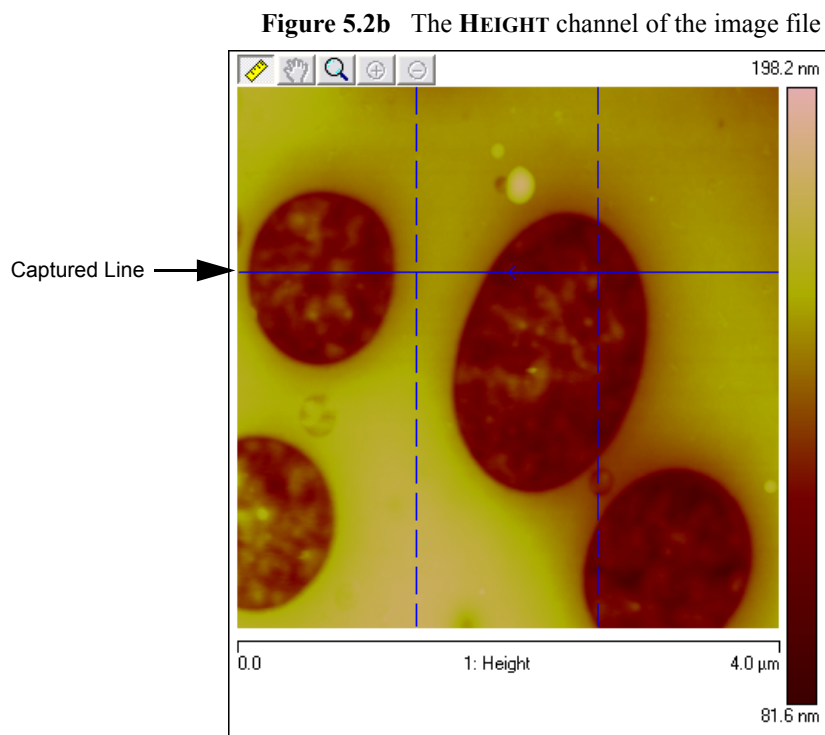
- Click the QNM-Hsdc Force Curve-Image icon to open the **QNM Hsdc Force Curve-Image** window, shown in [Figure 5.2a](#).

**Figure 5.2a** The QNM Hsdc Force Curve-Image window



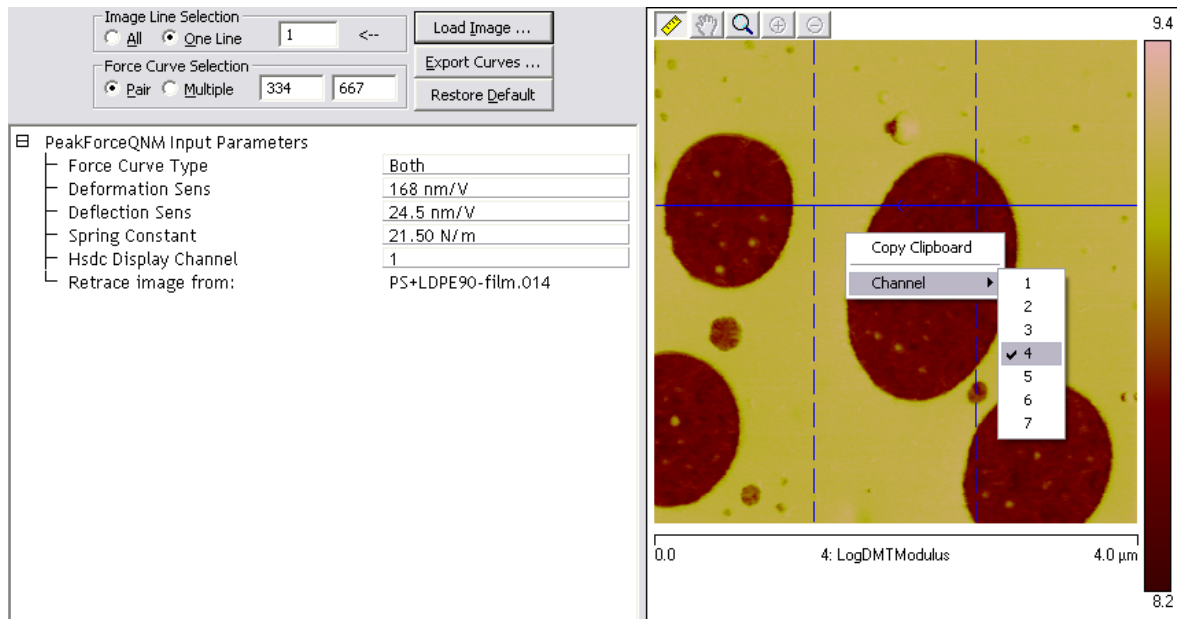
- Click the **LOAD IMAGE** button, circled in [Figure 5.2a](#), and select the image file associated with your high speed data capture file.

5. The solid blue horizontal line, shown in [Figure 5.2b](#), displays the captured line.



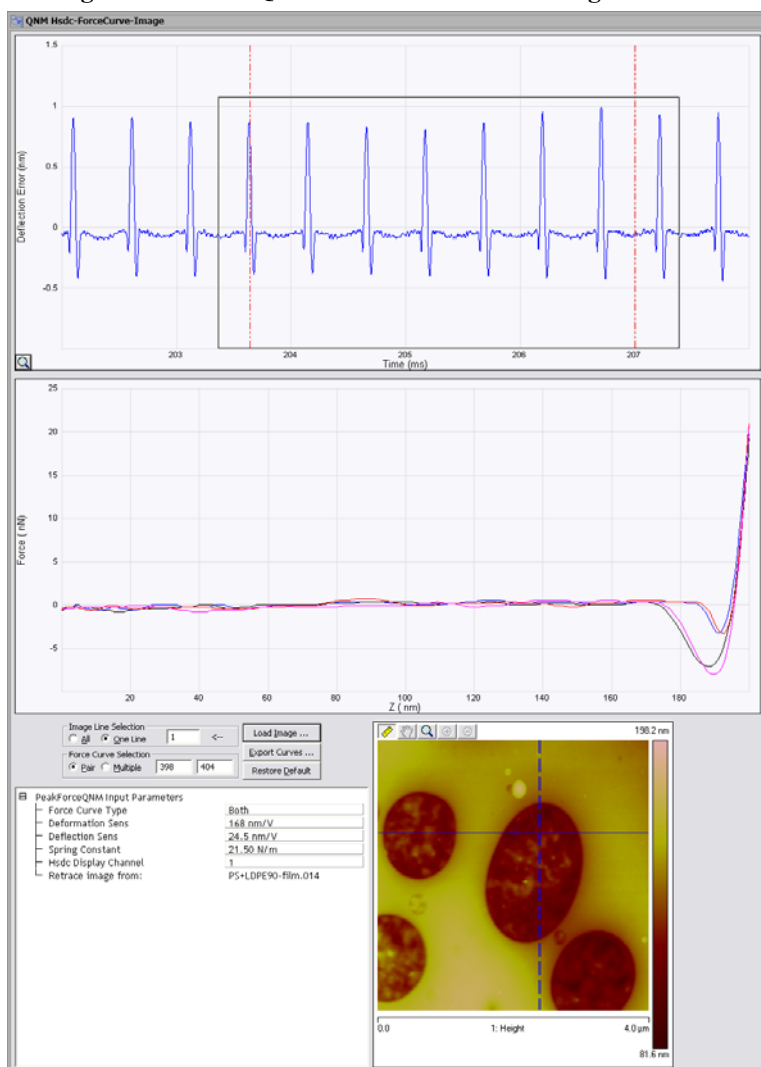
- Two vertical dashed blue cursors, shown in [Figure 5.2c](#), display the X position of the displayed force curves when **PAIR** is checked in the **Force Curve Selection** box. The associated number boxes display the Z piezo tap number. You may move the force position by either dragging the dashed blue cursors in the image, the dashed red cursors (see [Step 7](#)) in the time display or entering the numbers in the **Force Curve Selection** panel. You may select the channel of the captured image by right-clicking in the image as shown in [Figure 5.2c](#).

**Figure 5.2c** Vertical cursors display X position



7. The position of the force curves is also represented by the red dashed cursors in the **Deflection Error vs. Time** display, shown in [Figure 5.2d](#). To Zoom in on an area of interest in the graphs, hold down the Control key and draw a box in the preferred area. To Zoom back out, double-click the image or click the magnifying glass icon in the lower left corner of the plot.

**Figure 5.2d** The QNM Hsdc Force Curve-Image window



## 5.3 Controls and Settings

### 5.3.1 Image Line Selection

*Range and Settings: ALL:* Displays all captured lines.

**ONE LINE:** Displays one captured scan line of taps. The arrow in the **Image Line Selection** panel and the solid blue line, shown in [Figure 5.2b](#), display the scan direction. The counters are displayed in the **Force Curve Selection** panel.

**Figure 5.3a** PeakForce QNM Controls

Image Line Selection  
☐ All ☒ One Line 0 -->

Force Curve Selection  
☒ Pair ☐ Multiple 461 462 -->

Load Image ...  
Export Curves ...  
Restore Default

PeakForceQNM Input Parameters

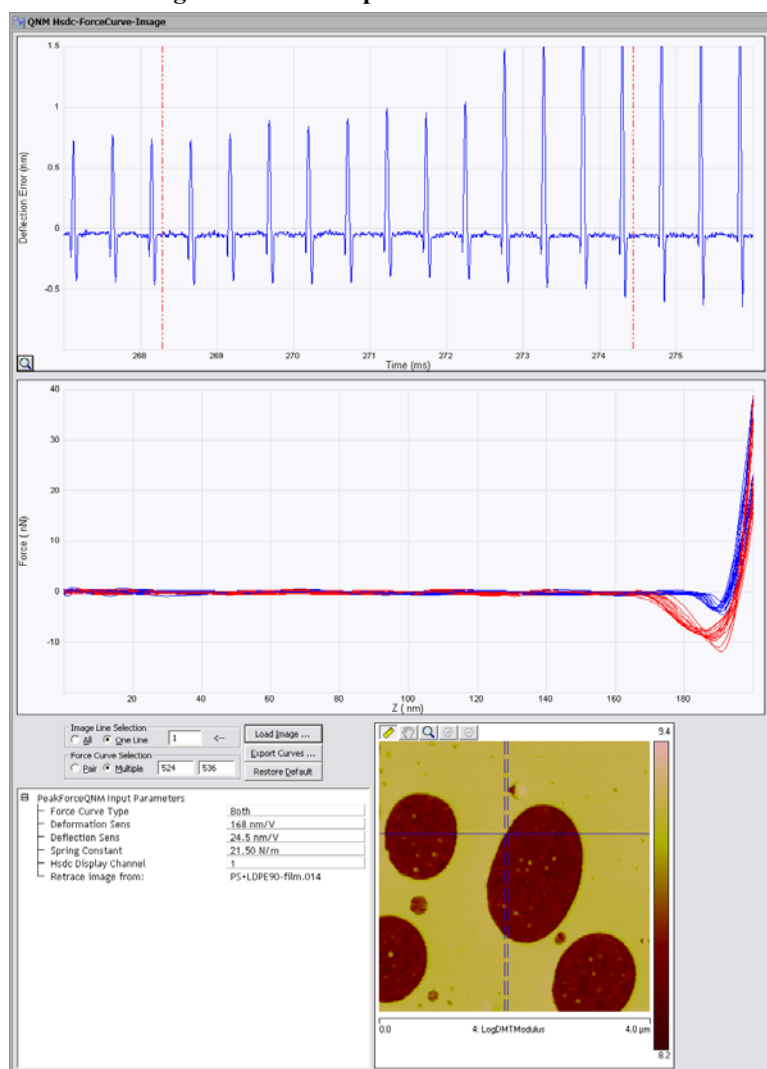
- Force Curve Type: Both
- Deformation Sens: 172 nm/V
- Deflection Sens: 45.0 nm/V
- Spring Constant: 33.33 N/m
- Hsdc Display Channel: 1
- Retrace image from: tesp-ps-ldpesp.001

## 5.3.2 Force Curve Selection

**Range and Settings: PAIR:** Displays a pair of force curves. See [Figure 5.2d](#) for an example. The location of the pair of force curves can be controlled by moving either the dashed blue cursors in the image, the dashed red cursors (see [Step 7](#) on [Page 73](#)) in the time display or entering the numbers in the **Force Curve Selection** panel.

**MULTIPLE:** Displays all the force curves between the vertical cursors. The location of the force curves can be controlled by dragging either the dashed blue cursors in the image, the dashed red cursors (see [Step 7](#) on [Page 73](#)) in the time display or entering the numbers in the **Force Curve Selection** panel. See [Figure 5.3b](#).

**Figure 5.3b Multiple Force Curve Selection**



### 5.3.3 Force Curve Type

*Range and Settings:* **BOTH:** Display both the extend and retract portions of the Z piezo ramp.

**TRACE:** Displays the extend portion of the Z piezo ramp.

**RETRACE:** Displays the retract portion of the Z piezo ramp.

### 5.3.4 Exporting Force Curves

Click the **EXPORT CURVES** button to export either a **PAIR** of force curves or **MULTIPLE** curves in a directory FrcExport. One file will be created for each force curve. This binary file (header is ASCII) can be opened by the NanoScope Analysis package.

Because off-line plots (either NanoScope or NanoScope Analysis) feature more display options for exported curves (FrcExport) than HSDC files, you may wish to export your curves and then open the FrcExport files. Details can be found in the *Line Plot and Multiple Line Plot* page of the *Force Curve and Ramping Analysis* in the help pages of the NanoScope Analysis package.

## 5.4 PeakForce QNM Input Parameters

The parameters appearing in the **PeakForce QNM Input Parameters** window have been collected in real-time but may be modified for off-line analysis here.

- Deformation Sensitivity
- Deflection Sensitivity
- Spring Constant
- Hsdc Display Channel

## 5.5 Exported Force Curves

Exported force curves (file names begin with FrcExport) can be viewed in NanoScope software or NanoScope Analysis software. Off-line plots feature more display options for exported curves than are available for HSDC files. These additional options are listed below.

## 5.5.1 Time Domain Plots

Change the **X Data Type** from **Z HEIGHT** to **TIME** to transform the original force curve, shown in [Figure 5.5a](#), to a **Force vs. Time** plot (a.k.a. heartbeat), shown in [Figure 5.5b](#).

**Figure 5.5a** Exported force curve

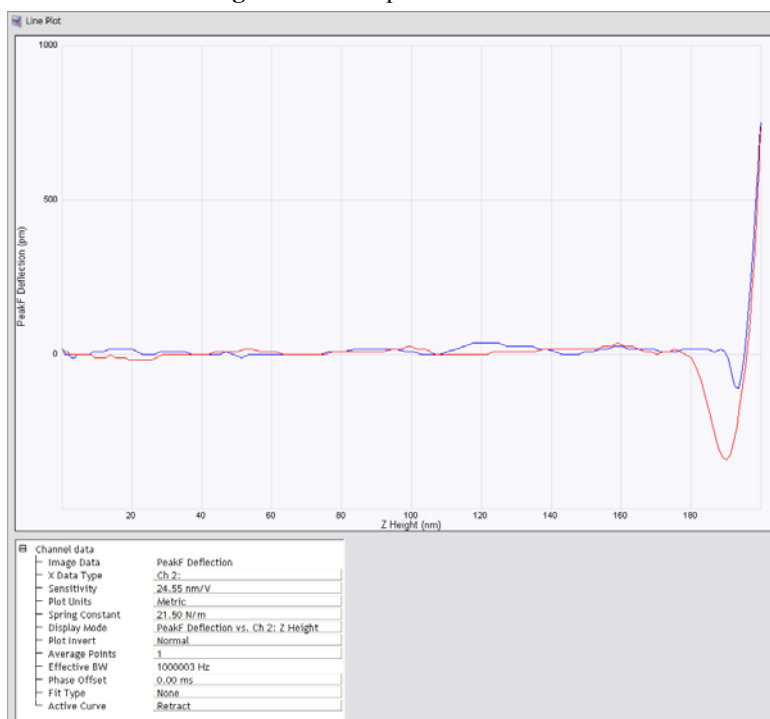
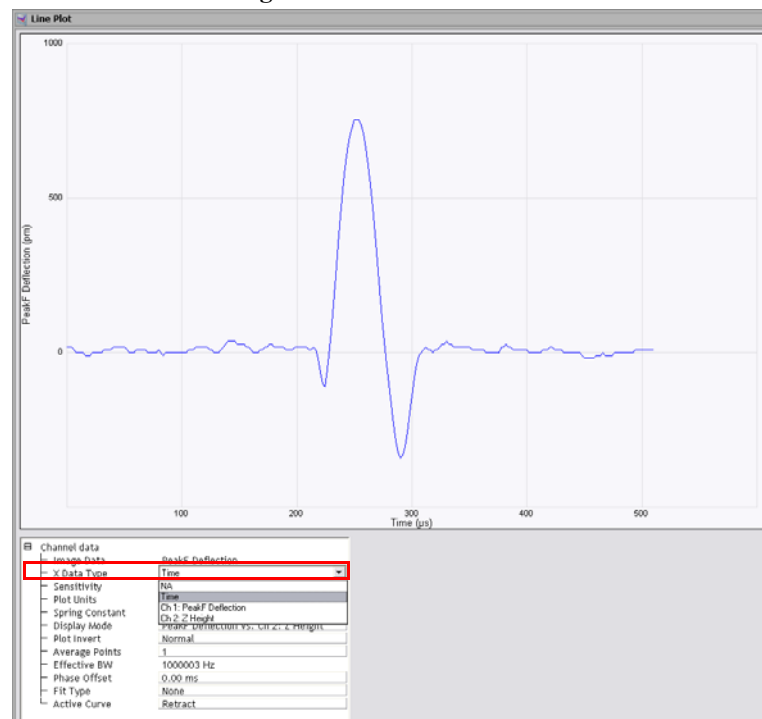




Figure 5.5b Force vs. time



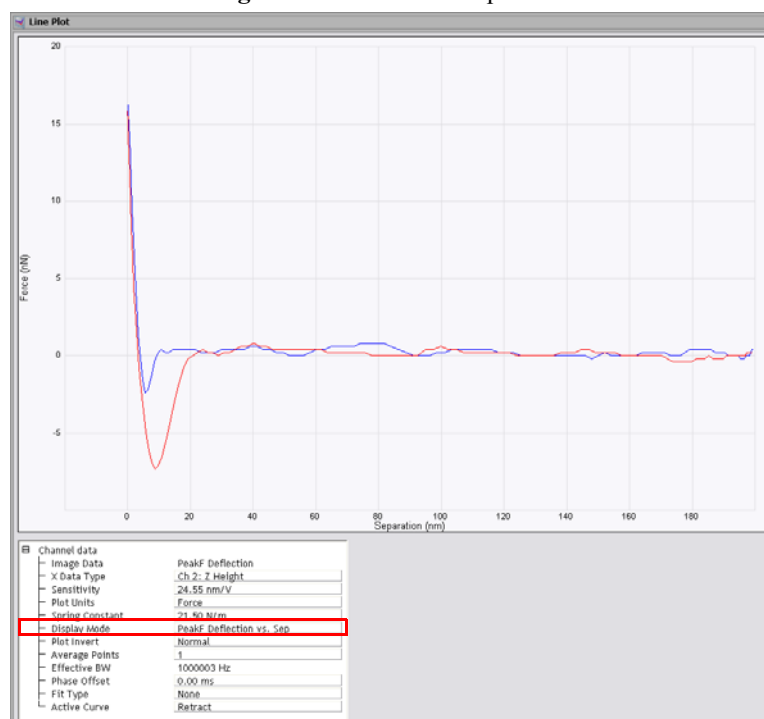
## 5.5.2 Plot Units

The Y axis can be displayed as **VOLTS** (Deflection), **METRIC** (distance in nano meters) or **FORCE** (nano Newtons).

### 5.5.3 Display Mode

Change the **Display Mode** to **PEAKF DEFLECTION VS. SEP**, shown in [Figure 5.5c](#), to plot your data vs. separation.

**Figure 5.5c** Force vs. separation



## 5.6 Image Math

You can use the **IMAGE MATH** functions in NanoScope Analysis software to re-evaluate your results if you wish to change a parameter.

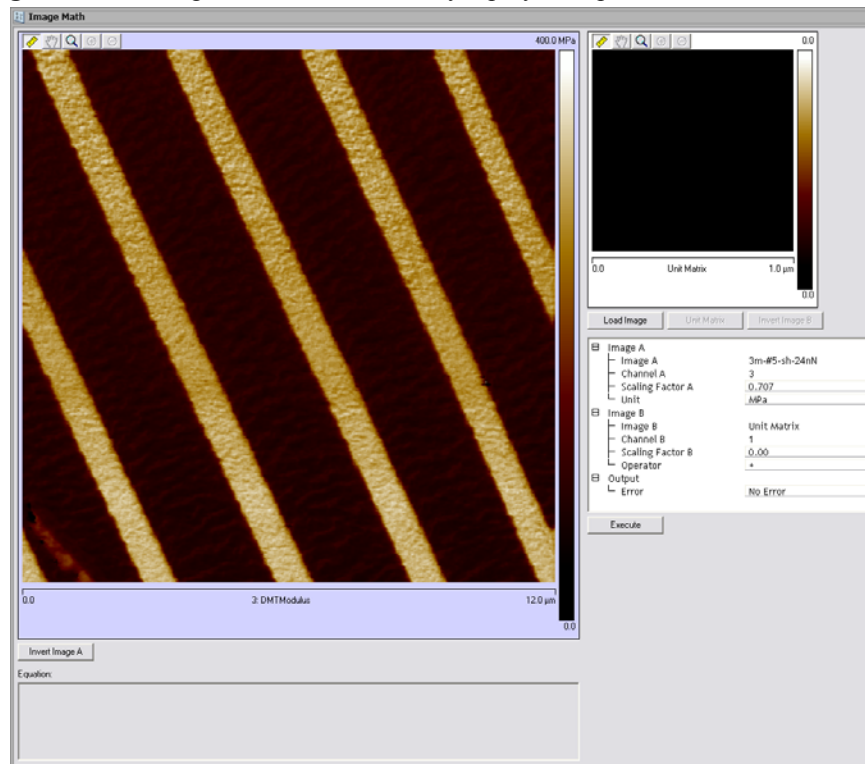
For instance, you can re-compute Young's modulus if you wish to compensate for a changed tip radius or a different spring constant.

Because

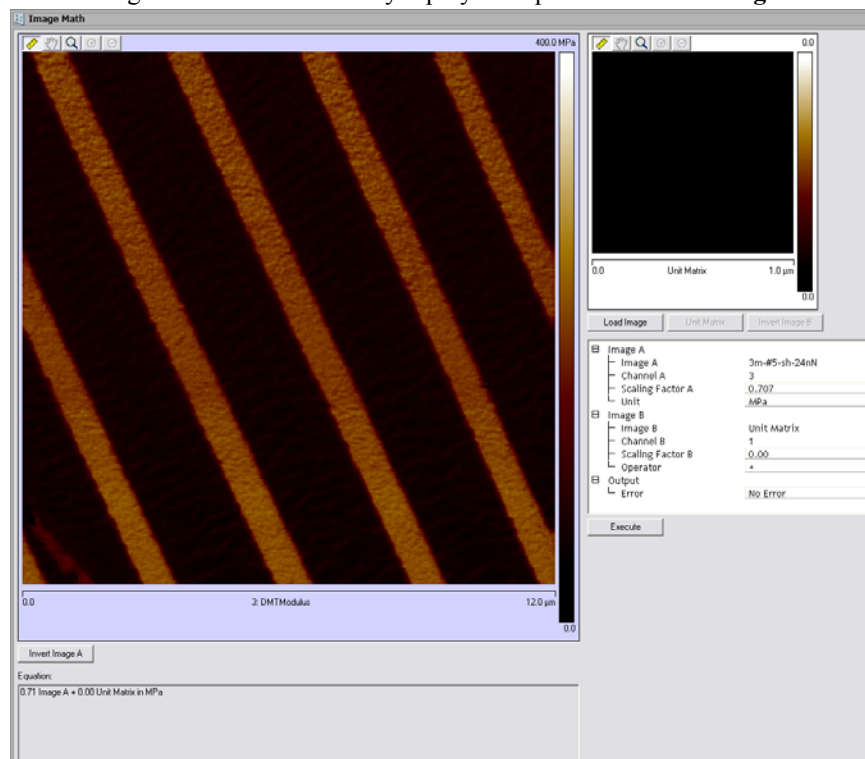
$$E \propto \frac{k}{\sqrt{R}}$$

you can scale Young's modulus by to arrive at corrected results. [Figure 5.6a](#) shows an image of Young's modulus in a multilayer polymer optical film before correction. [Figure 5.6b](#) shows an image of Young's modulus in a multilayer polymer optical film after multiplying it by  $(1/\sqrt{2})$  to compensate for a tip radius that has increased by a factor of 2 (e.g. from 10 nm to 20 nm).

**Figure 5.6a** Young's modulus in a multilayer polymer optical film before correction



**Figure 5.6b** Young's modulus in a multilayer polymer optical film after **Image Math** correction



The **Image Math** interface is shown in [Figure 5.6c](#) and the corresponding equation appears in [Figure 5.6d](#).

**Figure 5.6c** The **Image Math** interface

Image A	
Image A	3m-#5-sh-24nN
Channel A	3
Scaling Factor A	0.707
Unit	MPa
Image B	
Image B	Unit Matrix
Channel B	1
Scaling Factor B	0.00
Operator	+
Output	
Error	No Error

**Figure 5.6d** The **Image Math** equation

Equation:
0.71 Image A + 0.00 Unit Matrix in MPa



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